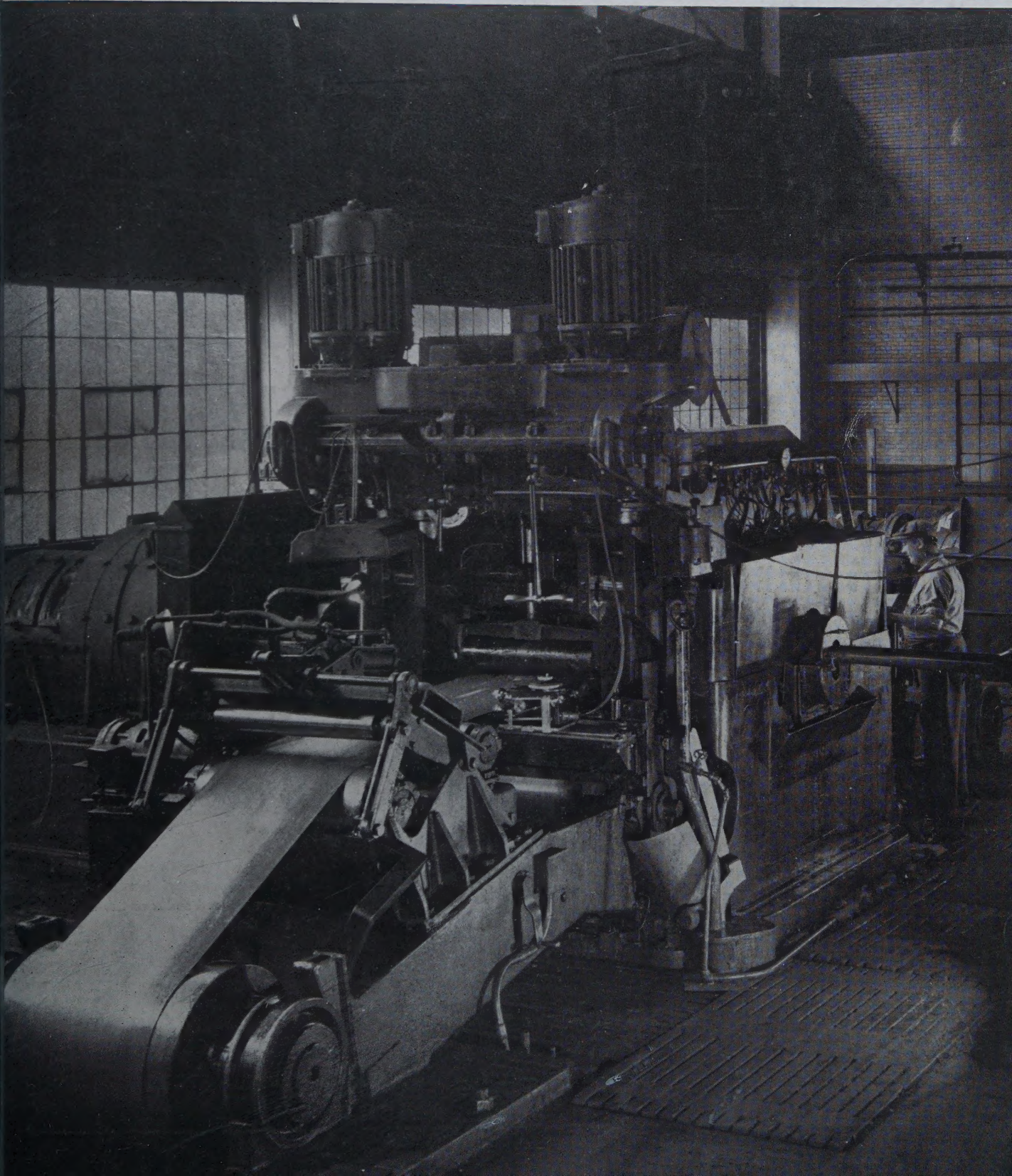


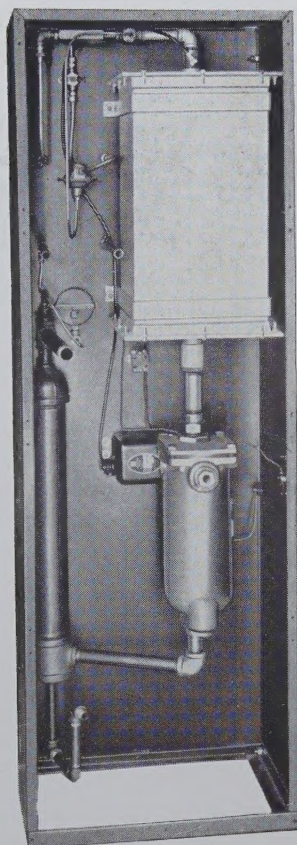
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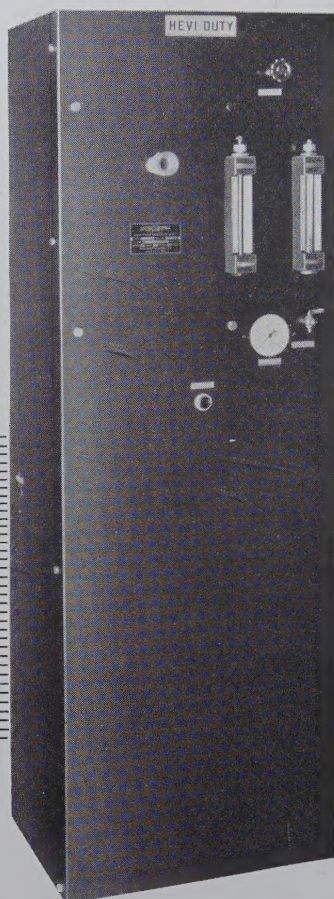




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VOL. I, NO. 9

SEPTEMBER 1949

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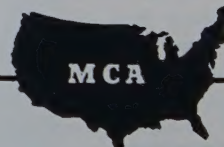
Published the fifth day of each month by the American Institute of Mining and Metallurgical Engineers, Inc., 29 West 39th Street, New York 18, N. Y. Telephone: Pennsylvania 6-9220. Subscriptions \$8 per year for non-AIME members in United States and North, South and Central America; \$9, foreign; \$6 for AIME members, or \$4 in combination with a subscription to *Mining Engineering* or the *Journal of Petroleum Technology*. Single copies, 75 cents; special issues, \$1.50. . . . The AIME is not responsible for any statement made or opinion expressed in its publications. . . . Copyright 1949 by the American Institute of Mining and Metallurgical Engineers, Inc. . . . Registered cable address, AIME New York . . . Indexed in Engineering Index, Industrial Arts Index and Chemical Abstracts . . . Entered as second-class matter Jan. 12, 1949, at the post office at New York, N. Y., under the Act of March 3, 1879.

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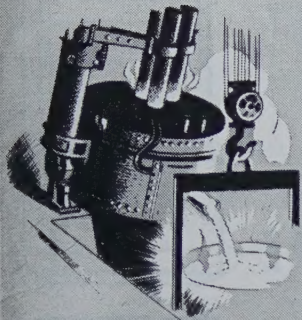
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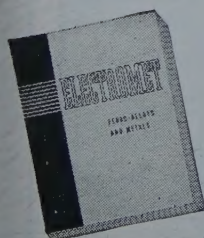
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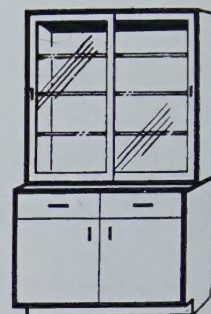
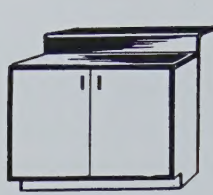
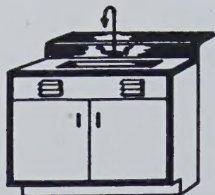
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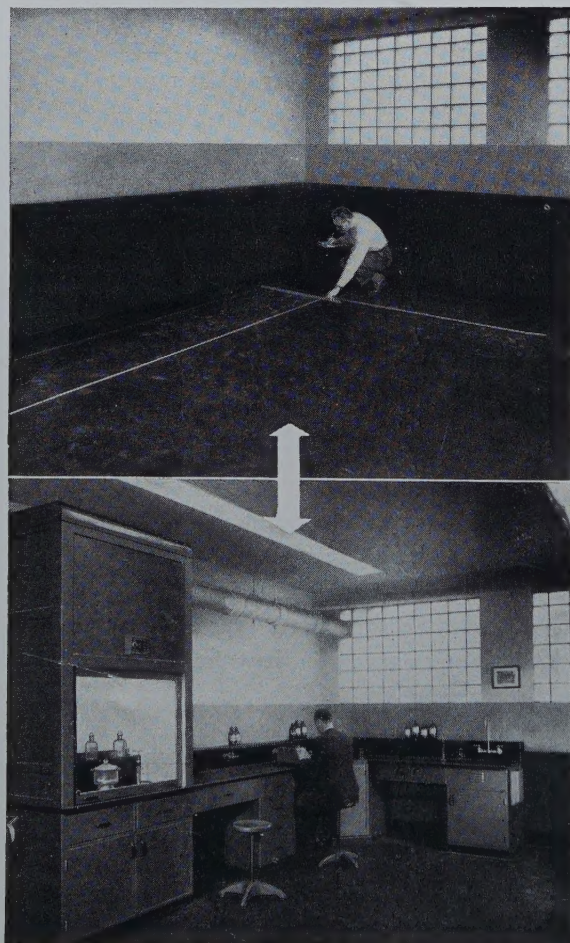
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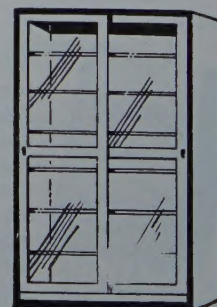
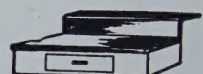
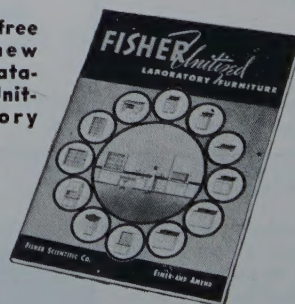
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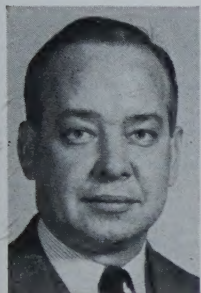
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D. F. Hilty (p. 20): Born Apollo, Pa. Attended Apollo High School and Penn State, graduating with B.S. in Electrochemical Engineering. Member of AIME and now lives in Niagara Falls, N. Y. He has worked for Carnegie Illinois Steel Corp. Was 1st Lieutenant in the Corps of Engineers, U. S. Army, and now research metallurgist with Union Carbide and Carbon Co.

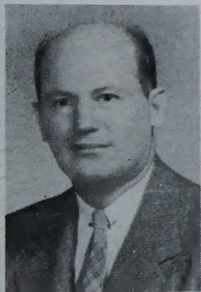
Hobbies are fishing and photography. . . . **Max J. Spendlove** (p. 553); born in Provo City, Utah. Went to Bingham Canyon High School and took his B.S. in E.E. in 1936 from the University of Utah. Now lives in Takoma Park, Md. Has worked at U. S. Bureau of Mines on the application of sonic and ultra-sonic waves; also research work on electrolytic and carbothermic production of magnesium at Salt Lake and on vacuum distillation of metals at College Park, Md. Besides writing technical papers, Mr. Spendlove's extra-curricular activities include photography, fishing and reading. . . .

John Wulff (p. 561); born in New York City. Graduated from Colorado School of Mines, Yale University and Tuebingen, Germany, with degrees of E.M., M.S., Dr. Sc.



J. Wulff

Is a member of AIME, and now lives in Cambridge, Mass. Was Research Fellow Munich University 1929-31 and since then Professor of Metallurgy, M.I.T. Mr. Wulff has previously presented many AIME papers on Corrosion, Powder Metallurgy, etc. . . . **Pol Duwez** (p. 571); born in Mons, Belgium. Attended Belgium high school and went to School of Mines, and University of Brussels, Belgium, with degree of Metallurgical Engineer 1932 and Doctor in Science 1933 (Physics). Now lives in Pasadena, Calif. Is a member of AIME. Was Professor School of Mines, Mons, Belgium, Research Engineer California Tech; is head of the Material



M. Cohen

Section Jet Propulsion Lab., and Associate Professor of Mechanical Engineering at California Tech, in Pasadena. His major hobby is music. . . . **Morris Cohen** (p. 578); Born in Chelsea, Mass. Went to Chelsea Senior High School and graduated from M.I.T. with degrees of S.B. and Sc.D. Is a member of AIME and now lives in Swampscott, Mass. Was Assistant Professor of Physical Metallurgy, 1937-46, and since then Professor of Physical Metallurgy at M.I.T. . . . **D. J. Blickwede** (p. 578): Born in Detroit. Graduate of Colet High School and Wayne University with B.S. degree and from M.I.T. as Sc.D. Is a member of AIME and now lives in Washington, D. C. Has worked as Metallurgist for Curtiss Wright Corp., research assistant M.I.T., and now is head of High Temperature Alloys Branch, Naval Research Lab., Washington, D. C. His recreation is fishing and watercolor painting. . . . **Howard T. Clark, Jr.** (p. 558): Born in East Hampton, Conn. Went to East Denver High School, Denver, Colo. Graduated from University of Denver with degrees of B.S.C.H.E., and M.E. (Phys. Chem.) and with degree of M.E. (Metallurgy) from Yale. Is a member of AIME and now lives in Stratford, Conn. Was Instructor in Chemical Engineering. Univ. of Denver; senior engineer, Denver Ordnance Plant, and now research metallurgist, Remington Arms Co., Bridgeport. Mr. Clark has previously presented an AIME paper as co-author on the "Constitution and Properties of Copper-rich Copper-chromium and Copper-nickel-chromium alloys." . . . **A. B. Greninger** (p. 590): Born in Glendale, Oregon. Went to Instituto Inglés, Santiago, Chile. Attended Stanford and Harvard Universities. Has B.S. Engineering and Sc.D. degrees. Is a member of AIME and now lives in Richland, Washington. He has been on Harvard engineering faculty, and worked with General Electric Co. in Cleveland, Chicago. . . . **C. S. Barrett** (p. 599): Born in Vermillion, S. D. Gradu-



H. T. Clark



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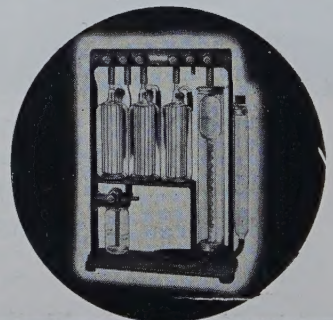
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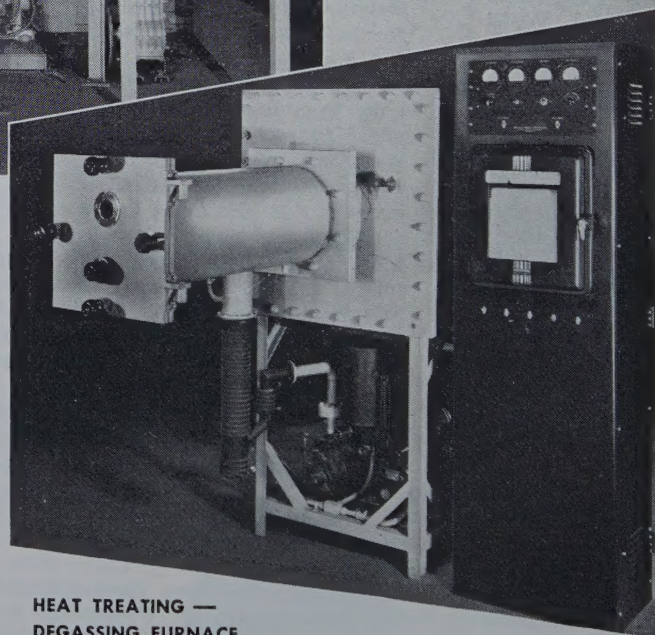
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ated with degree of B.S. from University of South Dakota, and Ph.D. from University of Chicago. Is a member of AIME and now lives at Flossmore (suburb of Chicago). He has been on the staff of U. S. Naval Research Laboratory, Washington, D. C.; in the Metals Research Lab. and Department of Metallurgy, Carnegie Tech, and Institute for the Study of Metals, University of Chicago. Mr. Barrett has previously presented AIME papers on researches on structure of metals and on physical metallurgy. His interests include tennis, water colors and suburban gardening. . . .

J. B. Hess (p. 599): Born in Bloomsburg, Pa. Went to Bloomsburg High School and graduated with degree of B.S. from M.I.T.

Is a member of AIME and now lives in Chicago. He has worked with J. H. Williams & Co., Buffalo, as junior metallurgist, in the Magnesium Div. of Dow Chemical Co., and as metallurgist on the staff



C. S. Barrett

of Institute for the Study of Metals. His special interests are gardening and swimming. . . . **A. G. Guy** (p. 607): Born in Chicago. Attended John Marshall High School and went to University of Chicago, graduating with degree of B.S.; from Ohio State with degree of M.S. and D.Sc. degree at Carnegie Tech. Now lives in Raleigh, N. C. He has worked with Wyman Jordon Co. as chemist; with Wisconsin Steel Co. as openhearth observer, and as research engineer, General Electric Co., and is presently Professor, Mechanical Engineering Dept., North Carolina State College. His major activities are bicycling, gardening and bird watching. . . . **E. P. Klier** (p. 611): Born in Flora, Ill. Went to Washington (Ind.) High School. Attended University of Notre Dame, graduating with degrees of B.S., M.S., Ph.D. Is a member of AIME and now lives in State College, Pa. He is Assistant Professor, Pennsylvania State College. Mr. Klier enjoys athletics, fishing and hunting. . . .

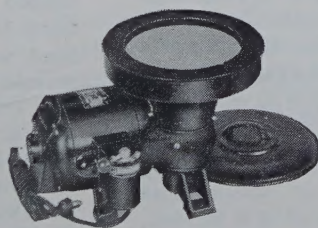
S. Marguerite Grymko (p. 611): Born in Kingston, Pa. Went to Kingston and Hanover Township High School. Attended Temple University and Pennsylvania State College graduating with B.A. and M.S. (Metallurgy) degrees. Now lives in Columbus, Ohio. Miss Grymko has previously worked at Hamilton Standard Propeller Co. as Asst. Metallurgist; at Pennsylvania State College, as research assistant, and at present at Battelle Memorial Institute, as research engineer. Miss Grymko's interests include golf, tennis, swimming. . . . **R. L. Dietrich** (p. 621): Born in Wheeling,



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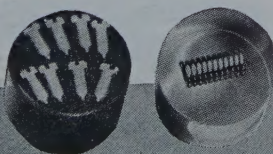
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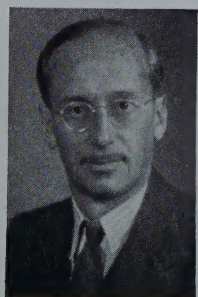
W. Va. Went to Warwood High School, Wheeling. Attended Carnegie Institute of Tech, graduating with B.S. degree. Now lives at Midland, Mich. He is working as metallurgist at the Magnesium Laboratory of Dow Chemical Co. His interests include photography and gardening. . . .



R. L. Dietrich

tendent of Metallurgical Lab. His major activities besides writing technical papers are golf and swimming. . . .

W. R. Hibbard, Jr. (pp. 635, 655, 598 and 620): Born in Bridgeport, Conn. Went to Crosby High School, Waterbury, Conn. Attended Wesleyan University, graduating with degree of B.A.,



P. Beck

and Yale University with D.Eng. degree. Is a member of AIME and now lives in New Haven, Conn. He has presented a great many AIME papers. His home dominates his interests and he is an amateur photographer. . . .

Y. C. Lin (p. 635): Born in Shanghai, China. Attended National Chiao-Tung Univ., China, and Yale Univ., graduating with degrees of B.S. in M.E. Now lives in New Haven, Conn. He has worked as a trainee in National Highway Administration. He is interested in driving,

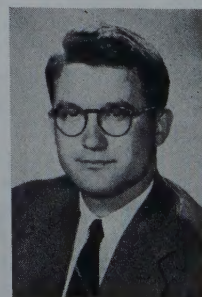
fishing, collecting of stamps and likes to travel. . . . **S. F. Reiter** (p. 635): Born in New York. Went to Townsend Harris High School. Attended Cornell University graduating with degree of B.S. in Mech. Engineering, and Yale with degree of M.S. in Metallurgy. Pursuing studies for Doctor's degree at Yale. Served in Navy during the War, graduating from Radio Materiel School, Naval Research Lab., Washington, D. C. Now lives in New Haven. He has worked for Chase Brass & Copper Co. as tool engineer. His favorite recreation includes music, painting and riding horseback. . . .



W. R. Hibbard

J. R. Low (p. 637): Born in Washington, Pa. Went to Oak Park High School, Illinois. Attended Purdue University graduating with degree of B.S. and Carnegie Tech with degree of D.Sc. Is a member of AIME and now lives in Ballston Lake, N. Y. Has worked for Keystone Steel & Wire Co., and Republic Steel Corp. as metallurgist. Was research assistant and Graduate Fellow in Met., Carnegie Institute of Technology, Asst. Professor of Metallurgy, Pennsylvania State College, and now Head, Metallurgy Research Section, Knolls Atomic Power Lab., General Electric Co., Schenectady. Mr. Low has previously presented AIME papers on aging and yield point in steel, and plastic flow of aluminum aircraft sheet under combined loads. . . .

R. I. Jaffee (p. 646): Born in Chicago. Went to Hirsch High School. Attended Illinois Institute of Technology Harvard University and University of Maryland, with degrees of B.S. S.M., Ph.D. Is a member of AIME and now lives in Columbus, Ohio. He has been research metallurgist at University of California, also for Leeds & Northrup Co., and Battelle Memorial Institute as assistant supervisor. His recreation includes fishing and golf. . . . **I. E. Campbell** (p. 646): Born in Kenton, Ohio. Attended Evansville College and Ohio State University, graduating with A.B. and Ph.D. degrees. He has worked for Sunbeam Elec. Mfg. Co., as experimental engineer and now is assistant supervisor, Div. of Nonferrous Metallurgy of Battelle Memorial Institute. His home is in Gahanna, Ohio. Mr. Campbell enjoys fishing and tennis. . . . **R. Maddin** (p. 655): Born in Hartford, Conn. Went to Weaver High School (Hartford). Attended Purdue from which he graduated with degree of B.S., and Yale with Dr. Eng. degree. Is a member of AIME and now lives in Baltimore. He has been Research Fellow in Metallurgy at Yale University, and Asst. Professor of Mechanical Engineering at



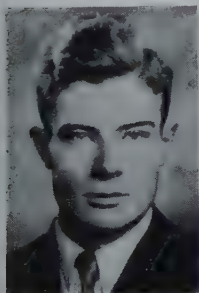
R. I. Jaffee

Johns Hopkins Univ. His major interests are sailing, fishing, tennis. . . **R. A. Chegwiddden** (p. 570): Born in Paterson, N. J. Went to Dover High School, N. J. Attended Polytechnic Institute of Brooklyn with degree of E.E. Now lives in Chatham, N. J., and is a member of technical staff of Bell Telephone Laboratories. His recreation includes music, fishing and bowling. . . **C. H. Mathewson** (p. 655): Born in Essex, Conn. Went to high schools in Milford and New Britain, Conn. Attended Yale University graduating with Ph.B. degree and Göttingen, Germany, with Ph.D. Is a member of AIME and now lives in New Haven. Has been at Yale University continuously since 1907, in the Department of Metallurgy. Prof.



R. Maddin

Mathewson has previously presented a large number of AIME papers. He enjoys travel. . . **Anna M. Turkalo** (p. 663): Born in E. Taunton, Mass. Went to Taunton High School and attended Boston University graduating with degree of A.B. Her home is in Schenectady, N. Y. Miss Turkalo has taught General Science in Junior High School, worked in the Welding Research Section, Watertown Arsenal, and at present is connected with the Knolls Atomic Power Laboratory of General Electric Co., Schenectady. Her hobby is mountain climbing and hiking. . . **D. Turnbull** (p. 663): Born in Stark County, Ill. Went to Kewanee High School. Attended Monmouth



D. Turnbull

College graduating with B.S. degree and University of Illinois with Ph.D. His home is now in Schenectady. He became Instructor and Asst. Professor of Physical Chemistry at Case Institute of Technology and later Research

Associate at the Research Laboratory of General Electric Co., Schenectady. Mr. Turnbull has previously presented an AIME paper on Transient Nucleation. He enjoys gardening, hiking, literature.

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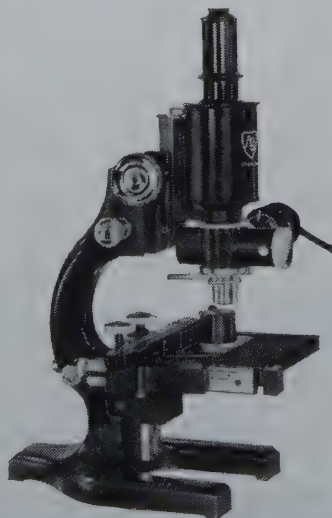


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* Titanium ingots weighing 65 lb for sometime have been made by Allegheny-Ludlum at Watervliet. Within several weeks 100-lb ingots will go into production and 400-lb ingots are planned by year's end. Allegheny-Ludlum has taken a very sizable order for large titanium forgings to be used in gas turbines. The company will today accept orders for wrought titanium in about any form, the price being on the order of \$20 a lb for strip. It is expected that this price may drop to a mere fraction of this level within several years--probably to a level competitive with stainless.

* Determination of oxygen in titanium, a long-awaited development, is possible by proper modification of the vacuum fusion analytical procedure as ordinarily applied to steels. G. Derge, of Carnegie Tech, will describe the procedure in detail next month.

* The world's largest openhearth will be lighted about Sept. 10 by Weirton Steel Co. at Weirton. Rated at 550 tons it probably will tap well over 600 tons. It will be all basic except for a silica roof and will fire either oil or tar (p. 31).

* Gaseous fluxes may prove invaluable for those critical applications in which conventional fluxing techniques are not applicable. Boron trifluoride is shown for first time (p. 25) to be particularly attractive. Joint quality is exceptional; there is no attack on the steel surface.

* Unalloyed titanium, titanium-hydrogen alloys and 0.25 at. pct oxygen alloys can be cold rolled 95 pct without edge-cracking. Higher oxygen alloys edge-crack at 60-70 pct and titanium-nitrogen alloys edge-crack at much lower reductions. (p. 654)

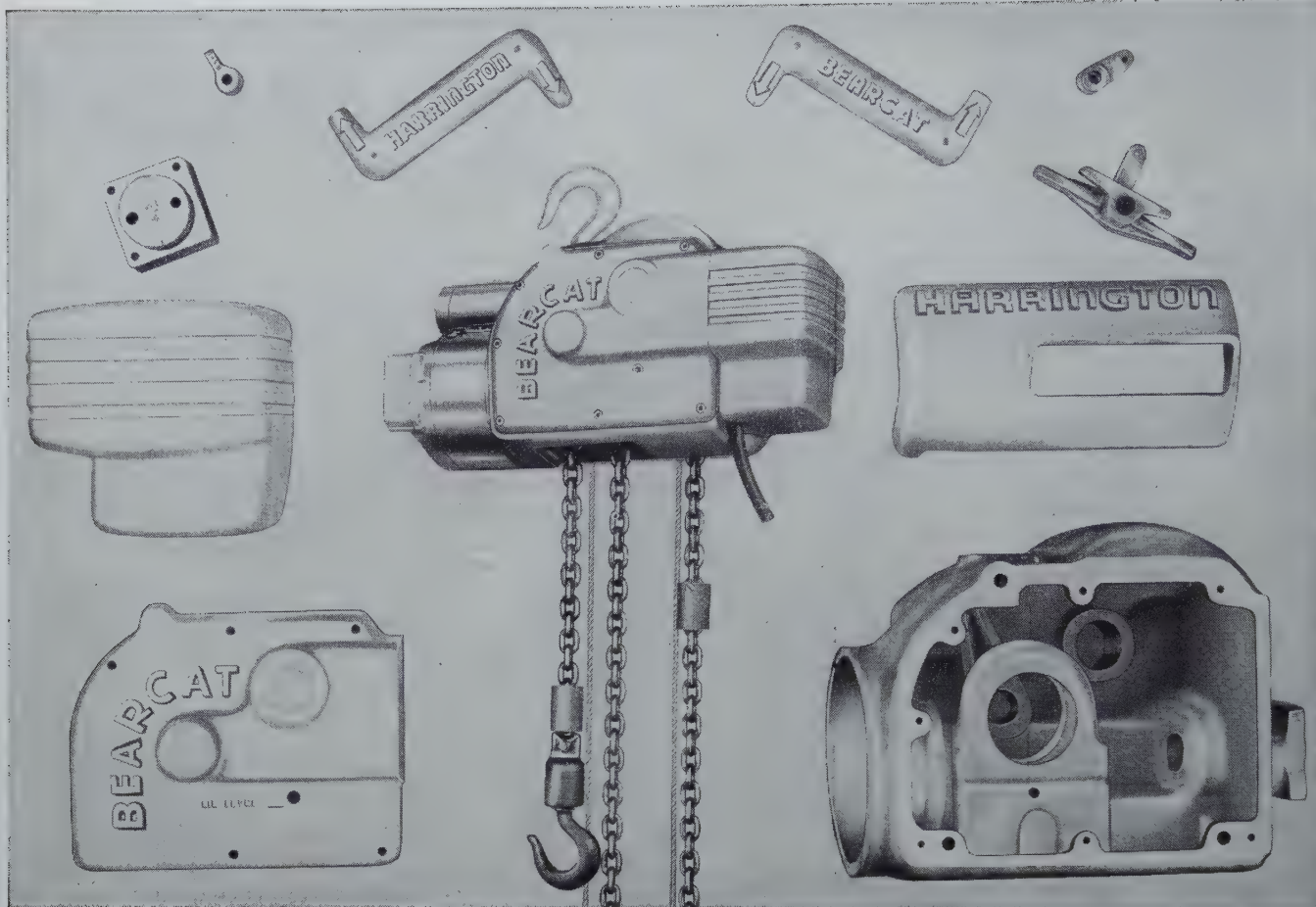
* Newport Steel Corp. is establishing encouraging records with its new 66-in. single-stand reversing hot mill. Twelve 13,000-lb ingots are being rolled into 36-in. wide, 0.109-in. strip each hour. Three ingots are working at one time. One is being roughed to 7/8 in., one breakdown is being reheated and one reheated breakdown is being rolled on reversing hot mill.

* In Japan, the slump in export sales following the world recession and the fixing of the yen exchange rate at 360 to the dollar has whet the appetite of Japanese manufacturers for markets in China. Communist leaders working under the large Soviet mission in Tokyo are effectively baiting the hook with promises of large markets in Communist China.

Japan would, for instance, like to stop importing Canadian coking coal at \$24 a ton and change back to Kailan (Chinese) coal at \$15 a ton. The changeover would enable Japan to export iron and steel profitably far below the present "floor price" fixed by General MacArthur's SCAP in order to prevent undercutting the West.

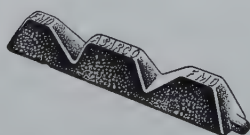
* Steel is no longer scarce in Germany now that production is up to $9\frac{1}{2}$ million tons a year. Fuel is likewise in excess supply with the exception of coking fines, and this shortage will disappear by Fall with new Ruhr capacity coming into production.

* The Ruhr Authority, established with great difficulty last year to force German exports to patch up the economies of other European countries, is now in an embarrassing situation. The other European countries have surpluses of their own and are fearful of Germany's reentry into world markets. Dr. Adenauer, the probable future Chancellor of Western Germany, is now calling the Ruhr Authority an instrument of commercial espionage to stifle German competition.



JMLco. P80

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Casting ten parts of the Bearcat electric hoist (above) in TENZALOY enabled the manufacturer, The Harrington Company, Philadelphia, to:

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METALS DIVISION

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Henry Kaiser

Says . . .



Introduction: As you know, Mr. Kaiser, the **Journal of Metals** is the technical magazine for the metals industry and its readers are engineering executives . . . what interests you will certainly interest them!

Henry J. Kaiser: Yes, I know . . . A. B. Ordway and Chub Rich and several other executives of Kaiser Steel have long been active in your steel group. George Vreeland of Kaiser Engineers, is a member. So's Tom Price. Dusty Rhoades [D. A. Rhoades, gen. mgr. of Permanente Metals Corp.], is with you, too.

Q: Incidentally, how's that aluminum racing boat of yours coming along?

A: Well, we're sure having our troubles. A well known boat-works did the designing and building . . . when we got the boat, we discovered that we only had started—the real work was still to be done. During the first trial runs, tremendous mechanical difficulties developed . . . the gear case bolts sheared off, the shafts had to be replaced to meet higher stresses, the designs in general had to be improved . . . the boat didn't perform properly under the varying water conditions and speeds that had to be met. Every boating enthusiast and engineer had a pet theory of his own on what to do, and hardly anyone agreed. We're aiming for record-breaking speeds so high that we must perfect all the details to get the performance the boat was originally designed for.

Q: Who's building the new shafts?

A: Allegheny-Ludlum.

Q: Will the boat be ready for next season's races?

A: Oh yes! Right now we're up to our ears in hull changes. She's too low in the water . . . she gets up and planes nicely, but it takes her too long to get up. We're widening the sponsons on the hull, 30 in. on top down to 6 in. along the bottom. That should get her up higher, and she'll get up on the step much faster . . . It certainly should correct all the troubles we've had with instability.

Q: What's the world speed record?

A: About 140 . . . well, to be exact, it's 141.74 mph.

Q: Will you beat that?

A: Yep! No question about it. It's just a matter of cramming in the power and making the proper hull and engine alterations. I'm pretty sure we may even push her above 150 mph.

Q: How's the business situation for the remainder of the year?

A: Most things are shifting on the up side. As you know, early in the year the whole economic picture went a little sour . . . everyone went around with a droopy look and people started to talk down

prices until they were blue in the face. But the manufacturer still had his 42¢ raw material dollar and 47¢ manufacturing dollar . . . prices were sweated down a bit, but there are limits to that. For example, the Kaiser car right now is selling too low for all the value in it . . . now there are definite signs that the readjustment period has about run its course and that an upturn is starting.

Q: How about 1950?

A: Good! That is, it'll be good if business gets off the dime and practices a little more free enterprise instead of talking all the time about it. Why, I was even getting into the habit until Mother [Mrs. Kaiser] told me a couple of months ago to stop talking and do something! One thing I'd like to do is in some way, somehow, help blast open the dam holding back the flow of equity capital moving into plant expansion. Luckman [Charles Luckman, president of Lever Brothers Co.] had the right idea when he said recently that we're only beginning—that in common sense, we have no excuse for a recession, let alone depression. We must show the gumption, courage and teamwork to gear the economy up so it will indefinitely employ everyone who can and wants to work.

Q: Well, your big building program at Fontana certainly shows confidence in the future!

A: Yep, and confidence in the Pacific Coast, too. We're completing \$35 million dollars worth of expansions at Fontana. The new blast furnace [with capacity of 1200 tons of pig daily] will double the basic hot-metal potential out there. We're really turning out plumbers' pipe—enough for one home every 30 sec. . . . And the opening of the Eagle Mountain iron ore mine puts our raw material picture in good shape for generations to come. The West's really boiling and we're going to be in there boiling with it.

Q: Are you going to name the new blast furnace after a granddaughter?

A: No. It'll be Bess No. 2, after Mrs. Kaiser.

Q: I see by the papers that you have made quite a deal with the government so Permanente Metals can take over all the aluminum facilities it has been operating on lease.

A: Yes, we're really in aluminum for the long pull. We're producing 20 pct of the nation's primary aluminum. The whole thing's an integrated unit from bauxite reduction at Baton Rouge . . . to fabricated cable at Newark, Ohio. We've opened a new foil plant. We have just purchased from War Assets three major aluminum plants for \$36,000,000. It's a good deal, and we're glad to own our own

home. The government is accepting 24 million pounds of aluminum pig from us to meet the down payment and interest on the plants.

Q: You mentioned a little while back that there was a need for more equity capital. What are your ideas along that line?

A: Well, history records show that ever since 1869, about every 20 yr, the United States has doubled its economy. Oh, there was one period during the depression of the 30's when this rate of growth in the gross national product was checked. But the historical pattern is for us to double national production every 20 yr. . . . Now, let's look at the future! Let's figure out a program about what our economy is going to be in the future . . . get our sights fixed and drive ahead in that direction.

Q: Could you make any estimate of what the future national income could be?

A: Sure. Let's set a goal of about \$300 billion by the year 1954. That's not an impossible figure. It would mean a lot more real goods for everyone in the country.

Q: Is more deficit financing and inflation the answer?

A: No! That's silly. It's a dead end. The only answer is more factories turning out more goods. Plenty more, at a steadily lower manhour cost.

Q: Well, what is the answer?

A: We had better limber up and unleash some dynamic plan to release a flood of investment by industry. According to Slichter [Dr. Sumner H., Harvard economist] we are billions of dollars on the short side in industrial plant and equipment. He states that we need to invest \$50 billions to get American industry back up to the level where it has as much invested per worker as it had in 1929. It's pretty startling, don't you think, that today there's one-fifth less capital invested per worker than in 1929? Imagine having less industrial capital per worker than 20 yr ago! Slichter recently told me that the nation needs new capital investments in industry totalling \$78 billion. Something will have to be done to enable industry, not government, to invest that capital. If industry doesn't do it, you can be darn sure that the government eventually will.

Q: Is there any way to encourage industry to make such heavy investments?

A: Yes sir! Rosenstiel [Lewis C.] is pushing an idea that makes real sense. Get the government to figure out some means of incentive taxation to encourage businesses to invest and permit them to write off their capital outlays in a much shorter time. And industry, in turn, would make some kind of a firm arrangement with labor that would provide an advance each year based on increase in productivity. This will have to be a long-range stabilized affair. It's suggested that productivity could be raised 4 pct annually and labor rates adjusted automatically upward . . . if some such agreement could be reached with labor, it might stop the wrestling match every year between labor and management.

Q: What do you mean by incentive taxation?

A: Well, you'll just have to stimulate risk capital—equity money—that is so badly needed if new jobs are to be created. There sure is one way to

stimulate these investments. It worked during the war. There was a law allowing faster amortization, or accelerated depreciation of capital investments. Any number of manufacturers had the opportunity to write off certain investments in 5 yr instead of the usual 20 yr. Well, you saw the result. The law stimulated \$6.5 billion worth of industrial investments. If it can work in war, why can't it work in peace?

Q: There seems to be a scattering of talk for a revised amortization procedure!

A: Yeah! But some of the reports seem to give the impression that business is trying to get out of taxes and load the cost of government onto somebody else. That's bad! Somebody has to pay the taxes and industry certainly should pay its share. But new tax plans could be devised that would encourage the businessman but still in the end increase the revenue flowing to the government. Let businessmen charge off their new equipment in 5 yr and you'd see such a spurt in new equipment and construction it would make your eyes bulge. Slichter has another good idea—devise a rebate on the income surtax for savings that the taxpayer plows back into building up the industrial and business life of the country.

Q: How could you get a program like that across?

A: Get down to the grass roots! There's too much advertising of free enterprise by one businessman to another. You must get the idea down to the smallest hamlets, and every little city. Maybe you could devise an organizational committee, a temporary committee. There would be representatives at every crossroad. They would carry the crusade for the program. They'd get over the idea that this isn't just something for big business . . . it's something that's going to benefit everybody tremendously. The whole thing will feed on itself . . . create continued prosperity . . . better than the country has ever hoped for. The only way this can ever be done is with constant investment by industry in machinery to turn out more and better goods. Not a couple of years' spurt in investment and then a dry period, but a heavy flow of capital year in and year out. I think it can be done. Rosenstiel is entitled to great credit for his effort to get across his plan for sustained prosperity. I don't want to be a prominent factor in the campaign, but I'll do anything I can to carry on the program. I'm willing to be a laborer in the effort.

Q: Do you have any comments on the recent Steel Industry-CIO hearings?

A: Well, no. It's just what I'm talking about, however. Everybody is trying to split the same pie into more pieces. It can't be done. Industry is going to want to have more steel, and steel has got to build more and more efficient capacity if the workers are going to get higher real wages over the long pull.

Q: What do you think the trend of steel prices will be?

A: They certainly won't go down. They'll go up, if anything. The long term trend will be up until we get the benefit of what I have already described . . . greater and greater capital investment for more efficient machinery and for greater productivity. And the public will benefit, too.

Q: How about the national debt? Is it ever going to be reduced?

A: Yes and no. That is, the national debt won't be a serious burden—it can be reduced—if we do the positive things necessary to keep the economy always expanding. Why we could even get up to a \$500 billion a year level in the next 20 yr or so if we really made a united national drive to accomplish it. The only way we will spend all this money is to create it . . . not create it by deficit financing but by production . . . that's the sound way. I don't see how anybody can quarrel with this plan that I have mentioned. As soon as the idea gets across to the grass-roots and hamlets, and people understand the principles of the whole thing, it could go through Congress like a breeze. People are pretty smart. They'll recognize the soundness of the whole thing.

Q: Some time ago, Mr. Kaiser, you made some comments on the role of younger people in industry.

A: Well, the old people may get discouraged sometimes, but younger ones aren't. Maybe the young people need a little direction and need to see a well-devised and challenging plan. It is up to enlightened industrial and business leadership to define this plan, or else government will define it for them . . . if young people see the plan with worthwhile goals, they will carry it out. Nothing will stop them. They have been through a lot and most of them have a brighter sparkle in their eyes than the oldsters. Of course, youth is a state of mind sometimes rather than actual age, and I'm thinking about proposals that would inject the vigor, imagination and courage of youth into shaping the nation's destiny . . . just the other day I flew up to Burlington, Vt., to have dinner with a friend of mine. He's a conservative. He seemed to be pretty concerned about his 13-yr old boy. The kid, according to him, was a liberal. The Dad sent him to a nice conservative school to tone him down. I kind of thought he ought to be tickled to death. At least the boy was thinking for himself, and from my conversation with him he seemed to be doing pretty well. It takes new ideas and fresh spirit in everything we do.

Q: How are things going in the automobile business?

A: Competition's really here again. . . . The auto industry must put everything its got into selling. . . . We licked the battle of production, and have sold upwards of 400,000 cars. . . . But now our number one problem is to get our selling forces into stronger and stronger position for the buyers market that's here . . . price reductions aren't the solution. We cut the price of our lowest priced car by \$333 to invade a new price field and to advertise "the lowest priced big car in the world." Now the main efforts are directed at helping strengthen and rally our dealers into tremendous efforts, and we're working on that aggressively.

Q: The Kaiser-Frazer Traveler seems like a real smart idea!

A: Yes, it's one of the greatest advances we've made . . . and the public appreciates it. We've been able to originate the postwar styling, the big roomy car with the widest seats yet developed, the high compression engine, the steel-top convertible—these are some Kaiser-Frazier contributions.



" . . . let's stop talking and start doing!"

Q: How about the one-piece diecast door designed by you and Doehler-Jarvis?

A: Well, that's a research job. It's interesting.

Q: Will the door be used next year?

A: Oh no!

Q: Do you have any particular idea as to how to increase car sales?

A: We are making a real study of establishing a lot of small assembly plants around the country. We'll soon have one or two working. All the hand-work on a car can be done just as well away from Detroit.

Q: Do you think there is any market in this country for cars like the British MG or the Jaguar?

A: No. Everybody thinks that. We gave the whole problem a great deal of study. A small tricky car catches on with only a small group of people.

Q: How about some safety factors that can be put into an automobile in case of accident?

A: Not if you are thinking of tricky ideas like a rubber-lined box for you to jump into in case you are about to have a crash . . . imagine having time before an accident to jump into a box. It's a question of engineering the whole car for better safety or roadability . . . making changes like narrowing the post at the windshield to minimize the blind spot, seeing that the car is perfectly balanced, that the brakes are right. . . . We've made great strides in these directions, and we keep on working on it.

Q: Any final observations, Mr. Kaiser?

A: Some of these things I have been talking about are very close to my heart. The United States is dangerously short of having enough productive capacity to meet the needs of the people. Human wants are limitless . . . as limitless as the imagination and daring of the American mind. Let's stop talking and start doing!

It's Everyone's Business

TODAY, over half way through 1949, recovery in Western Europe looks less certain. The broad political lines of cooperation, which converged upon the Atlantic Pact, have emerged relatively unscathed from the Senate debate. The attitude of many Senators, however, has shown that not all American leaders are committed to the definite abandonment of isolation. At the same time, the American recession has set unforeseen obstacles in the way of world recovery, not only by causing a decline in American purchases abroad but by inducing many Congressmen to believe that economy in national expenditure must include sharp cuts in foreign aid. The grand strategy of the western world is thus now neither as clear nor as firm as it was a year ago.

The most ominous of the clouds on the horizon continues to be the British dollar crisis, which is becoming somewhat of a political football on both sides of the Atlantic. Fateful indeed will be the Washington meetings next week between American officials and Britain's Cripps and Bevin. The British don't have a plan of merit to conquer their export deficit. As for the Americans, it is likely they will hint tactfully—or not so tactfully—at the possibilities of devaluing the pound and retreating from Socialism. Mr. Cripps will be quick to defend Socialism and undoubtedly will show the following table from the **London Economist**:

EXPORTS AND IMPORTS BY VOLUME
First Quarter of 1949

	Exports	Imports	Export Index divided by Import Index
Belgium-Luxbg....	114	97	118
Denmark.....	84	107	79
France.....	127	106	118
Norway.....	91	109	84
Sweden.....	65	103	63
Switzerland.....	113	121	94
United Kingdom..	156	82	190

His attitude certainly will be that Britain's current dollar difficulties are not related to the specific Socialist measures that have been taken and that a non-Socialist government would not have taken. The Americans are likely to remain quite unmoved, and a rather unhappy situation is in prospect.

Elsewhere on Capitol Hill the O'Mahoney Bill to legalize freight absorption and the use of delivered prices by businessmen **acting independently**, has sped through both Houses, and is getting a little ironing out of differences in conference. Sponsored by two famous trust busters, it is designed to reassure businessmen who feared that the Supreme Court decision in the cement and, especially the rigid conduit case outlawed the absorption and averaging of freight costs, a very traditional and widespread practice. Industries such as steel, which abandoned delivered pricing after the cement decision (and when demand was so high that consumers

could be induced to pay the freight charges themselves) are likely to resume absorbing freight charges where it is necessary to fight for orders. Recent increases in freight costs may, however, dampen their enthusiasm, and any return to an organized system of basing points is remote. Furthermore, as time progresses, the precise legal meaning of "acting independently" may provide the stuff of much expensive and exasperating litigation.

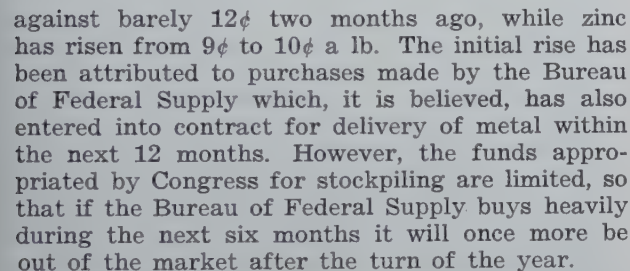
The O'Mahoney Bill, if it finally passes, will make a permanent amendment to the anti-trust laws. The greased way this Bill has slid through the Committees is due to the sponsorship of Senator O'Mahoney and Mr. Celler, both of whom have broken many a lance against the trusts. It might well be that their sympathies would lie on the other side but for the firm representations of beet-sugar growers and the infant chemical industries in Senator O'Mahoney's state of Wyoming and the strong case outlined to Representative Celler by the grocery chains. There is no denying that Representative Celler has been a busy man indeed, this hot summer in Washington. Part of the day it has been the O'Mahoney bill and the remainder of the day he has pushed hard on his bill to put additional "teeth" into the Anti-Trust Code.

Turning from Washington to the industrial mid-west, the business community is deriving some crumbs of comfort from minor shifts in sentiment, but the crumbs are still small and quite crumbly. Nonetheless, the prevailing mood is in distinct contrast to the gloom prevailing no more than a few weeks ago.

Industrial production, though still patchy, is far more encouraging than in June. Production, as measured by the Federal Reserve Board's index, fell for eight consecutive months and hit a new low point in July. August likewise may show a moderate decline but there are high hopes that September will be the turning point. Expenditures for plant expansion have held up well in the first half of the year, according to Government estimates. Granted this is so, the sharp drop in the Reserve index indicated a very severe cutback in consumer spending. It is now popular to assume that with price cuts already made or about to be made, that there will be an increase in inventory accumulation and consumer spending over the Fall months which should stimulate industrial output. But as an offset, Government estimates indicate a moderate decline in plant expansion expenditures over the remainder of the year. Despite all the weighty pronouncements of economists and such, no one is yet sure whether the year end will witness a further sharp decline in business activity or a moderate recovery. Certainly the recent buoyancy in the two very sensitive economic barometers, steel-making scrap and stock prices, is smoothing the furrows in many a brow. If this buoyancy persists the sound of laughter again may be heard in the Duquesne Club in Pittsburgh and the Brown Palace in Denver.

METAL PRICES
March 1 to August 15, 1949

Month	Electrolytic copper	Lead	Zinc
March 1	23.5	17.5	17.5
April 1	18.5	15.5	15.5
May 1	16.5	11.5	10.5
June 1	15.5	11.5	8.5
July 1	17.5	14.5	10.5
August 1	15.5	11.5	8.5



Labor's demands for further packets of money and security are likely to disturb the industrial picture for some time to come. A prolonged steel strike might well have sucked the American economy, now floating uneasily in economic cross-currents, into a whirlpool of depression. It was the fear of being held responsible for this catastrophe that finally drove the bitterly protesting steel companies to accept a 60-day truce while the President's fact-finding board went to work. Among the consuming industries, the automobile industry, while talking of strikes, has been almost alone on the peak of industrial prosperity.

The accompanying map shows that it is only in predominantly agricultural states that unemployment has not increased since last January. According to the Federal Security Agency, nearly half of the country's 98 largest manufacturing areas have over 7 pct of their labor force unemployed, compared with the national average of 6 pct. There are a number of districts where unemployment is up to, or over, 12 pct and five of these "depressed areas" are in New England. This explains why Boston was the Secretary of Commerce's first stop on the tour of the nation which he made in order to find out where pump priming is most urgently needed. It also explains Mr. Dewey's recent announcement of plans to start a series of public works in New York State. The President has ordered all government hands to stand by at the pumps in areas of acute unemployment. If necessary, efforts are to be coordi-

Appropriations to prime the pump are limited, however. If necessary, there may be enough water to dampen the present limited deserts of depression, but there is far from enough to prime all the pumps that would be needed if a drought really threatened the whole nation. Off in the wings, however, Senator Murray is touching up a permanent irrigation scheme. His Economic Expansion Bill of 1949 is intended to ensure a national income of \$300 billion, with all the spending and taxing such an objective implies. So far it's just a cloud as big as a hand on the horizon but it's big enough already to make any number of people very grumpy indeed.

High-Cr Steel in Acid Arc Furnace

by D. C. Hilty

In a recent publication¹, it was shown that the limiting equilibrium controlling the amount of chromium that can be retained in a steel bath for any given carbon content is expressed by the equation,

$$\text{Log } K = - \frac{15,200}{T} + 9.46$$

in which T is the absolute temperature and

$$K = \frac{\% \text{ Cr}}{\% \text{ C}}$$

Studies of basic arc-furnace practice, both at the Union Carbide and Carbon laboratories and in the field, have indicated that this equilibrium is approached quite closely in commercial basic furnaces. In the manufacture of high-chromium steels by the basic electric process, the amount of charged chromium that remains in the bath at the end of the oxidizing period is a direct function of the carbon content of the bath and the bath temperature in accordance with the above relation.

It has been considered probable that the relation also holds for acid arc-furnace practice. Up to the present time this belief has been unconfirmed, because of the lack of pertinent data from acid chromium-steel heats. Since it is becoming increas-

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¹"Relation Between Chromium and Carbon in Chromium Steel Refining," by D. C. Hilty, *Journal of Metals*, Feb. 1949, p. 91.

ingly evident, however, that the chromium-carbon-temperature equilibrium may be of fundamental importance, both technically and economically, in the commercial production of chromium steels, a check of the validity of the relation in the case of acid practice appeared desirable.

Through the excellent cooperation of the Cooper Alloy Foundry, Hillside, N. J., such a check has been secured. A visit was made to this plant for the purpose of making observations of the chro-

mium-carbon-temperature relation at the end of the oxidizing period in high-chromium steels melted in an acid-lined arc furnace. During the course of the visit, several heats were witnessed, and data from a number of others were obtained. Moreover, three experimental heats were made to explore the possibilities of improving the recovery of chromium in acid practice. These included the deliberate charging of excess carbon in order to protect charged chromium from oxidation during the early stages of a heat, the use of aluminum to reduce oxidized chromium from the slag, and the use of ferrosilicon and lime additions to reduce oxidized chromium from the slag. The details are reported below.

General Description of Practice: All observations were carried out on 1500-lb heats of 0.10 pct max carbon, 18-20 pct chromium, 10 pct nickel steel for castings. The furnace was an acid-lined, three-phase arc furnace having a nominal melting capacity of approximately 1500 lb per hr. Actual capacity of the furnace, however, was over 4000 lb of molten metal.

The usual melting practice employed for heats of this type is as follows: The furnace is charged with practically 100 pct chromium steel scrap consisting primarily of gates, riser, and a variable proportion of turnings. Chromium content of the charge is estimated as 17.4 pct, and the carbon content is adjusted to about 0.20 pct. The charge is melted down in approximately half an hour. During the meltdown, a small quantity of lime may be added to improve slag fluidity. When the bath is completely molten, the temperature is raised until it is in excess of 3000°F. Injection of oxygen to remove carbon is then begun at a rate of approximately 165 cfm. Oxygen injection is maintained for a period of 2-2¼ min. The electrodes are raised at the start of the injection period, and power is not re-applied to the furnace for the rest of the heat.

At the conclusion of the oxygen blow, low-carbon ferrochromium, low-carbon ferromanganese, and 50 pct ferrosilicon, sufficient to meet the composition specifications, are added. Ordinarily, there is no attempt to reduce oxidized chromium from the

CHROMIUM and carbon content and bath temperature at the end of the oxygen blow show that the equilibrium relationships previously shown for basic arc furnaces are also true in acid arc furnaces. One of three trial heats in which aluminum was fed to the oxidized slag, gave an indication of increase in recovery of chromium from scrap charged. One heat, purposely melted in at higher carbon than usual, gave an indication of possible time saving. Ferro-silicon and lime were ineffective in reducing chromium from the acid slag.

slag. The additions are made as quickly as possible, the bath stirred, and the heat tapped. Elapsed time from the start of oxygen injection to tap is on the order of 8 min and total heat time varies between 45 and 55 min.

The total weight of cold additions, made at the end of the oxygen blow, is approximately 15 pct of the final bath weight. Apparently, the temperature rise during oxygen injection is of sufficient magnitude to insure rapid and complete solution of this relatively large quantity of cold alloys without excessive chilling of the bath. Tapping temperatures are on the order of 3100°F or more, even though no power is applied to the furnace during the finishing period of a heat.

Partial logs of the heats observed are with this article.

Temperature Observation: The temperatures reported on the heat logs were observed by sighting an optical pyrometer on the surface of metal in a well-slugged spoon. The pyrometer was a Pyro instrument having a scale calibrated on the basis of the conventional 0.4 emissivity factor for molten iron. This emissivity correction does not hold for high-chromium steels, or even for carbon steels at high temperatures. The observed temperatures, therefore, have been converted to true temperatures with the aid of the emissivity corrections given by Goller², and the corrected temperatures are listed on the logs in parentheses following the observed temperatures. All temperatures mentioned

² "The Emissivity of Molten Stainless Steels," by G. N. Goller, Trans. A.S.M., Vol. 32 (1944), pp. 239-250.

in the body of the report, however, have had the appropriate emissivity corrections made and are considered to be actual temperatures.

Observations of bath temperature at the end of the oxygen blow were made on two of the heats witnessed. These temperatures and the corresponding carbon and chromium contents were as follows:

Heat No.	Temperature, °F	Pct Carbon	Pct Chromium
45054	3265	0.079	12.15
45055	3340	0.083	12.65

According to the chromium-carbon-temperature chart, retention of 12 pct chromium in the bath at a carbon content of 0.08 pct should occur at a temperature of 3300°F, or midway between the two observations. The agreement appears to be well within the accuracy of the optical pyrometer in measurements of this type.

Although no other direct observations of temperature were available, the following carbon and

chromium contents at the end of oxygen injection were observed in the other heats witnessed:

Heat No.	Pct Carbon	Pct Chromium
45064	0.052	9.61
45065	0.10	12.15
45068	0.072	12.65

In addition, the following results from still other heats were reported by the Foundry:

Pct Carbon	Pct Chromium
0.031	8.40
0.056	7.28
0.076	10.20
0.031	4.30
0.043	6.03
0.080	12.78
0.087	14.38
0.084	12.75
0.066	13.58
0.053	11.57
0.070	14.86
0.062	13.45
0.074	14.10

The data, along with similar observations on basic heats of comparable size and composition, have been superimposed on a chromium-carbon-temperature chart, Fig. 1. It is evident that the chromium-carbon-temperature relation is the same for both acid and basic practice. The average temperature at the end of the oxygen blow is indicated to be approximately 3335°F for these small heats.

Effect of High-C Charge on Cr oxidation: In basic practice the presence of excess carbon in the charge frequently reduces the amount of chromium oxidized, presumably by protecting the chromium against excessive oxidation during the early stages of a heat when temperatures are far below those attained at the end of the oxidizing period. In order to learn if this same effect can be obtained in acid practice, extra carbon was added to the charge of heat No. 45065. The addition, consisting of crushed electrodes, was made when the bath was approximately half melted. It was calculated to increase the carbon content of the charge to 0.75 pct, but poor recovery was obtained, so that the bath at melt-down contained only 0.48 pct carbon, still more than double the usual amount.

Oxygen injection was started at a somewhat lower temperature (2890°F) than usual (3100°F). Within half a minute, a vigorous boil developed. The blow was maintained until the carbon flame subsided, but it was stopped immediately thereafter. The duration of the injection period was 2 min, 45

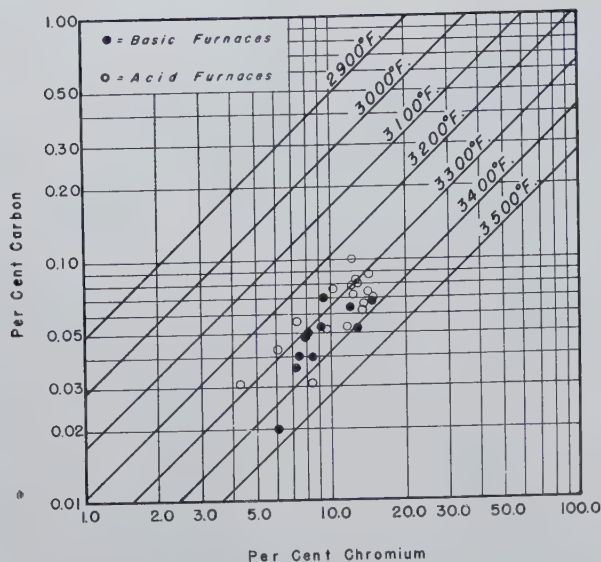


Fig. 1—Chromium-carbon-temperature relation at end of oxidizing period in chromium steel heats made by both acid and basic practice.

sec, or approximately 30 sec longer than for a heat charged with the usual amount of carbon. The heat was then finished in the normal manner.

At the end of the oxidizing period, the bath contained 0.10 pct carbon and 12.15 pct chromium. In comparison with normal practice at this shop, no reduction in the amount of chromium oxidized was observed.

It is possible, however, that this high-carbon charge practice may have resulted in a saving in furnace time. As mentioned above, oxygen injection was begun at a lower temperature and, therefore, earlier than on normally charged heats. This was possible because there was no danger of over-oxidizing chromium while the temperature was low. Consequently, the time usually employed for raising the bath temperature prior to oxygen injection was materially decreased. No direct estimate of the specific time saving that may have resulted can be made. It is notable, however, that this heat required only 49 minutes furnace time in spite of being charged with a high proportion of turnings that prolonged the charging and melting operations more than usual.

Reduction of Slag with aluminum: The use of aluminum as an agent for reducing oxidized chromium from the slag was tested in heat No. 45064. This heat was charged, melted, and oxidized by the normal practice, and the usual addition of low-carbon ferrochromium was made at the end of the oxidizing period. Following the ferrochromium addition, however, 7½ lb of aluminum was added to the slag. The heat was then tapped almost immediately.

Analyses of bath samples at the end of the oxidizing period, after the chromium addition, and at tap were as follows:

Sample	Pct Carbon	Pct Chromium
After oxygen blow	0.052	9.61
After ferrochromium	0.072	16.08
Tap	0.056	17.47

According to these results, it appears that the aluminum addition reduced sufficient chromium from the slag to increase the chromium content of the bath 1.4 pct. This inference is further supported by the fact that the chromium addition was calculated for a final chromium content of approximately 18.5 pct, based on a chromium content after oxygen injection of approximately 12 pct. Oxidation of this heat, however, was carried further than usual, so that the chromium content of the bath at the end of the oxygen blow was only 9.61 pct. Nevertheless, the final chromium content was only 1 pct low instead of 2.4 pct, as might have been expected if no reduction of the slag had occurred.

It is of interest that the 7½ lb aluminum addition made to this heat would be calculated to cause a rise of 1.51 pct in the chromium content of the bath, assuming that the oxide of chromium present in the molten slag is CrO as suggested by results on the chromium-carbon-temperature equilibrium.

Recovery of oxidized chromium from the slag by means of additions of ferrosilicon and lime, according to established procedures in basic practice, was attempted in heat No. 45068. Normal practice was followed up to the end of the oxidizing period. At the end of the oxygen blow, however, an addition of 40 lb of 50 pct ferrosilicon, mixed with 33 lb of lime, was made to the slag. Approximately 10 lb of lime had also been added earlier in the heat. The ferrosilicon was screened to a mesh size of ¾ in. or less. This addition was followed immediately by the usual additions of 166 lb of low-carbon ferrochromium and 10 lb of low-carbon ferromanganese. The usual final addition of 8 lb of 50 pct ferrosilicon was omitted. The bath was then stirred thoroughly, and the heat was tapped. All of the additions were made quite rapidly; only 4 to 5 min elapsed between the end of the oxygen blow and the tap.

Bath samples were taken in duplicate before oxygen injection, after oxygen injection but before

TABLE I. ANALYSES OF BATH SAMPLES FROM HEAT No. 45068 (Analysis by Cooper Alloy Foundry)

Sample	Per Cent					
	C	Mn	P	S	Ni	Cr
Before Oxygen	0.22				0.98	16.35
After Oxygen	0.072				0.62	12.65
At Tap	0.086				1.42	18.50
Ladle	0.086	0.43			1.42	18.60

Analysis by U. C. & C. Research Laboratories, Inc.

Before Oxygen	0.22	0.44	0.030	0.049	0.91	11.53	16.02
After Oxygen	0.072	0.16	0.028	0.044	0.80	12.30	12.27
At Tap	0.077	0.60	0.029	0.042	1.34	10.58	18.27
Ladle	0.074	0.62	0.024	0.042	1.38	10.59	18.25

TABLE II. ANALYSES OF SLAG SAMPLES FROM HEAT No. 45068 (Analyses by U. C. & C. Research Laboratories, Inc.)

Component	Sample		
	After Oxygen	At Tap	Ladle
Acid Soluble Chromium	6.52%	9.99%	12.66%
Cr ₂ O ₃	35.60	26.03	17.91
SiO ₂	38.02	41.80	46.94
FeO	10.73	7.93	6.87
Al ₂ O ₃	1.54	1.86	2.20
MnO	5.31	4.94	5.45
CaO	3.70	6.78	6.94
TOTAL	101.42	99.31	98.98

the additions, at tap, and from the ladle. One set was analyzed by the foundry, and the other was brought back to the U. C. & C. Laboratories for comparative analysis. Slag samples were obtained after oxygen injection, at tap, and from the ladle. The slags were analyzed by the Laboratories. The analytical results from all of these samples are given in tables I and II.

At 100 pct silicon efficiency the ferrosilicon addition was sufficient to reduce nearly all of the oxidized chromium from the slag. According to the above data, however, it is evident that practically no reduction was obtained. Calculations of a chromium balance indicate that the increase in the chromium content of the bath from the end of the oxidizing period to the ladle can be almost exactly accounted for by the ferrochromium addition. The changes in slag composition; i.e., decrease in total chromium content, increase in SiO_2 , etc. can be explained on the basis of dilution by the

lime and the portion of the ferrosilicon addition that was oxidized. Apparently, a little more than half of the added silicon was recovered in the bath. **Conclusions:** From these observations of acid arc furnace chromium steel melting, it is concluded that the chromium-carbon-temperature relation, expressed by the equation

$$\text{Log } K = \text{Log } \frac{\% \text{ Cr}}{\% \text{ C}} = - \frac{15,200}{T} + 9.46,$$

is valid in acid as well as in basic practice.

In experiments made to investigate possibilities of increasing chromium recovery in acid practice, no improvement was observed from the use of a high-carbon charge or from an attempt to reduce the slag by means of ferrosilicon and lime additions. Reduction of the slag by aluminum appeared to have some advantage. However, these results, derived from one heat for each practice, are not considered conclusive.

LOG OF HEAT No. 45054

Charge:

Stainless steel scrap and turnings (10 pct) = 1320 lb.

Meltdown:

Test No. 1: 0.14 C, 16.65 Cr, temperature = 3220°F (3070°F). Blew oxygen 2.2 min at 165 cfm. Carbon flame rose in 0.8 min and dropped in 1.9 min. Vigorous boil. Slag fluid at end of blow.

Test No. 2: 0.079 C, 12.15 Cr, temperature = 3450°F (3265°F). Added 166 lb. l.c. FeCr, 8 lb. 50% FeSi, and 10 lb l-c FeMn. Tap. Temperature = 3220°F (3070°F).

Test No. 3: 0.064 C, 18.10 Cr. Time of heat = 54 min. Time from start of oxygen to tap = 8 min.

LOG OF HEAT No. 45055

Charge:

Stainless steel scrap = 1320 lb, pig iron = 10 lb. 12:40 p.m. Power on.

1:13 p.m. Melted. Test No. 1, 0.24 C and 17.10 Cr; 10 min delay waiting for crane.

1:23 p.m. Test No. 2, 0.23 C and 16.55 Cr. Temperature = 3300°F (3135°F). Blew oxygen 2 min at 165 cfm. Carbon flame rose in 32 sec and dropped in 1 min 45 sec.

1:26 p.m. Test No. 3, 0.083 C and 12.65 Cr. Temperature = 3550°F (3340°F). Added 166 lb l-c FeCr, 10 lb l-c FeMn, 8 lb 50 pct FeSi. Additions completed in $\frac{3}{4}$ min. Stirred bath. Slag fluid.

1:29 p.m. Tap. Temperature = 3260°F (3095°F). Test No. 4, 0.080 C and 18.85 Cr.

Temperatures in parentheses are corrected for emissivity of molten chromium steel.

LOG OF HEAT No. 45064

9:35 a.m.—Power on.

Test No. 1 at melt, 0.18 C and 14.44 Cr. Temperature = 3200°F (3055°F). Blew oxygen $2\frac{1}{4}$ min at 165 cfm.

Test No. 2, 0.052 C and 9.61 Cr. Added 166 lb l-c FeCr.

Test No. 3, 0.072 C and 16.08 Cr. Temperature = 3290°F (3125°F). Added $7\frac{1}{2}$ lb aluminum.

Test No. 4, 0.082 C and 16.50 Cr.

10:10 a.m.—Tap. Test No. 5, 0.056 C and 17.47 Cr. Temperature = 3300°F (3130°F).

LOG OF HEAT No. 45065

Charge:

Stainless steel scrap and turnings (20 pct) = 1320 lb.

Crushed electrodes = 9 lb.

10:40 a.m. Power on.

11:00 a.m. Added the crushed electrodes.

11:20 a.m. Melted. Test No. 1, 0.48 C and 17.12 Cr. Temperature = 3000°F (2890°F).

11:23 a.m. Start oxygen blow at 165 cfm. Carbon flame rose in 32 sec and dropped in 2 min 45 sec. Stopped oxygen as soon as flame dropped.

11:26 a.m. Test No. 2, 0.10 C and 12.15 Cr. Added 166 lb l-c FeCr, 10 lb l-c FeMn, 8 lb 50 pct FeSi.

11:29 a.m. Tap. Test No. 3, 0.10 C and 18.35 Cr. Temperature = 3300°F (3130°F).

LOG OF HEAT No. 45068

Charge:

Stainless steel scrap and turnings (20 pct) = 1320 lb.

2:20 p.m. Power on. Added approximately 10 lb lime during meltdown.

3:06 p.m. Melted. Test No. 1, 0.22 C and 16.35 Cr. Injected oxygen for 2 min 15 sec at 165 cfm. Carbon flame rose in 40 sec and dropped in 2 min.

3:09 p.m. Test No. 2, 0.072 C and 12.65 Cr.

3:10 p.m. Added 40 lb 50 pct FeSi ($\frac{3}{8}$ in. or less) mixed with 33 lb lime. Followed immediately with 166 lb l-c FeCr and 10 lb l-c FeMn. Stirred bath with rod. Fluid slag.

3:13 p.m. Test No. 3, 0.086 C and 18.50 Cr. Tap.

"15-Minute Pig Iron"

A few months ago Maurice Duplessis, Premier of Quebec, Canada, announced that a new method for making pig iron had been developed at Titan Steel Corp., 30 miles south of Montreal, which was expected to have dramatic possibilities in the development of Quebec's iron resources. The inventor, 72-year-old, Wisconsin born, Walter E. Dudley, subsequently has issued some information on the process, which demonstrates that, with an ingenious and convenient set of equipment, a reaction which can be conducted on a laboratory basis can likewise be carried out on a somewhat larger scale. The scale so far is much smaller than a modest industrial process and there is justifiable question as to whether the teachings of the pilot plant can be applied economically to considerably larger equipment.

The operation has been described as follows: Crushed ore is placed in a hopper and from then on the operation is automatic. The ore is screened and fed to the pulverizer where it is reduced to a powder. It then goes to a classifier and if additional fineness is required is automatically returned to the pulverizer. A special flue collects dust arising from these operations and returns it to the scale where batches are automatically weighed.

Each batch is then transferred to a mixer, where additional materials are added, depending on the nature of the ore and the type of finished product desired. These additions include reducing agents, briquet binders, and fluxes.

After a pre-determined time in the batch mixer the batch is tripped and goes to the press. Here it is pressed into briquets and these are fed onto an endless belt. The briquets are about 12 in. diam. and about 1 in. long, and may be seen in the accompanying photograph.

The belt carries the briquets through a pre-heater, and overhead type oven which gradually raises the temperature to 1000° F.

The pre-heated briquets pass through the oven into a tilting-type electric furnace. The furnace electrodes are lowered and heat the briquets for from 30 sec. to 3 min. While the electrodes are off and raised for 1 min, the briquets flow into the furnace. That is, the arc operates for one minute during which time no briquets enter the furnace; the arc is shut off and the electrodes are raised for one minute during which time the briquets flow into the furnace.

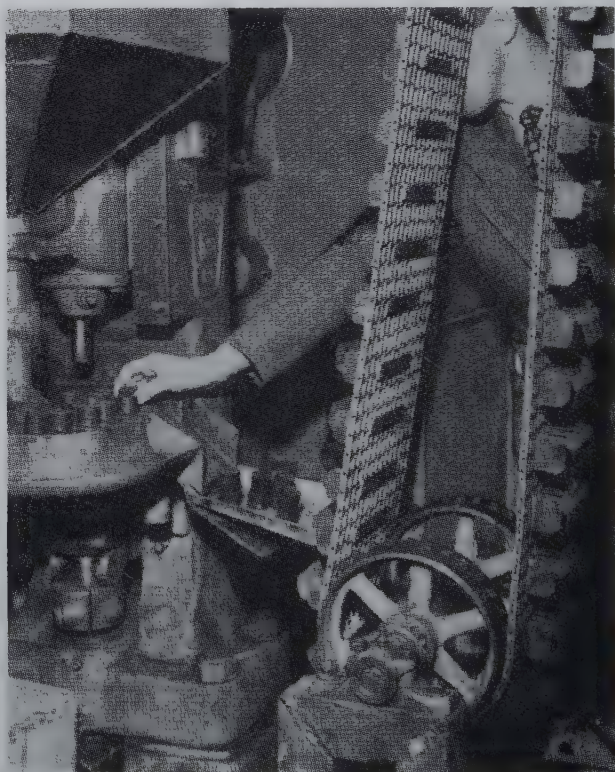
When the electrodes are down and delivering heat to the briquets a reaction is started which continues when the electrodes are again shut off and raised. Thus the briquets continue to release their own heat and the melting continues even

while the next briquets are dropping into the furnace.

The pilot plant now in operation delivers a 125-lb melt every 15 min or 500 lb an hr. The cost of this unit is given as \$250,000.

It is apparent then that if any one were interested in producing the tonnage of a 1000-ton blast furnace, approximately 42 of the Dudley units would be required, which would cost more than \$10,000,000.

No figures have been given on the cost of oper-



ation, current consumption, maintenance and repairs, percent reduction or recovery, and other critical items. Thus, any attempt to judge the economic merits of the process is futile.

Apparently low-grade ores are considered logical raw material for this operation. Assuming that at least two and one-half tons of ore would be required per ton of metal produced, the process would give rise to an enormous slag volume, all melted by electric heat, which is certainly not a particularly cheap fuel even in Canada.

Gaseous Fluxes for Brazing Steel

GASEOUS fluxes may prove invaluable for those critical applications in which conventional fluxing techniques are not applicable. Boron trifluoride is shown to be particularly attractive. Wetting and flow of molten brazing alloys are excellent, the quality of the brazed joint is exceptional, and most important, there is no evidence of attack on the steel surface.

• • •
by Alden P. Edson,

Donald G. Paquette and I. Laird Newell

• • •

QUITE understandably, there is no record of who first conceived the idea that it might be desirable to join together two pieces of metal in order to make a larger lump or a more imposing weapon. Very probably, however, that pioneer was considerably distressed by the obstinate refusal of many parts to adhere securely no matter how he heated and beat them or what metal he fused between them. Eventually he found that by propitiating the various gods and working solely in the proper phase of the moon while uttering appropriate incantations, some metals could be bonded in the solid state if certain strange reagents were present. Today the incantations and lunar phases have been discarded, but the strange reagents or "fluxes", first developed by the primitives are very much in evidence.

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Today's technician is painfully familiar with facts not available to the slightly more primitive metallurgist. It is known that most common metals oxidize immediately on exposure to air after even the most stringent cleaning operations, and that the rate of oxidation increases sharply as the temperature is increased. It is known that the demon which plagued the primitive man in his efforts to join metal parts was an oxide film which prevented intimate contact between metals and greatly impaired their adhesion.

There are several requirements placed on any material to be used as a flux for the joining of metals. It must react with and reduce or dissolve

the oxides and other contaminants normally present on the metal surfaces. It must protect the cleaned surfaces from further contamination during the joining operation. It must not unduly attack the metals themselves, but only their compounds. It must be displaceable from the metal surfaces sufficiently readily to permit intimate contact between the metals being joined. These criteria constitute a rigorously defined system, and it is indeed quite a tribute to the ingenuity and diligence of men that so many materials have been found which conform to all of these requirements.

By far the larger portion of the fluxes used for joining metal parts function in the liquid phase, depending for their cleaning action on the chemical solution of metal oxides in the molten flux, and permitting access of air to the cleaned surfaces only by relatively slow diffusion through the flux coating. Such reagents are necessarily highly active chemically, and usually exhibit some tendency toward attack on the metal surfaces during the joining operation. This is frequently deleterious to the quality of the finished assembly. However, often of greater importance is the problem of removal of flux residues from assemblies after completion of the joining operation.

There have been many unhappy experiences at

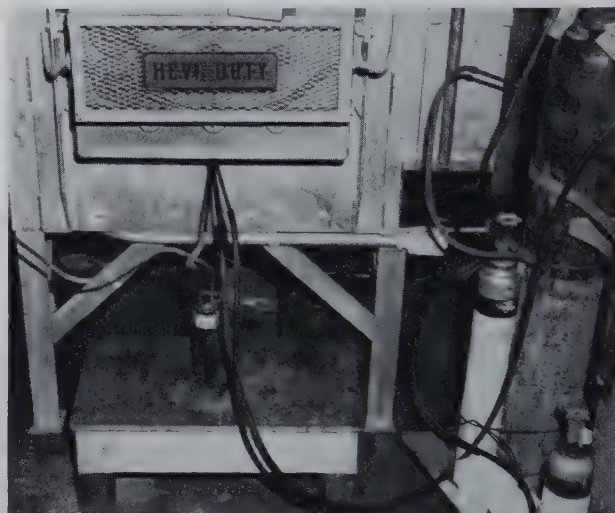
one time or another as a result of corrosion of joints which had been soldered with allegedly noncorrosive fluxes, or in parts on which a considerable effort had been made to remove potentially corrosive flux residues. There have been many unhappy experiences with parts from which the removal of flux residues constituted a major problem, and at times that problem assumes formidable economic proportions. The authors became involved in a problem in the latter category in brazing together the steel components of a vital and intricate highly-stressed assembly. After exhaustive tests of commercially available brazing fluxes for use in the assembly of this part, none were found which provided acceptable fluxing action at the desired temperatures but did not leave potentially corrosive residues. Because of the geometry of the part, which included several sharp and deep reentrant angles, complete removal of flux residues was most difficult. For this reason, an effort was made to develop a satisfactory flux which would leave no obnoxious residues. Work on new fluxes of conventional types yielded little promise of a remedy, so this project led naturally into fluxing in the vapor phase and the development of a number of agents suited for specific applications of this technique.

Vapor-phase fluxing—Several instances of flux applications in the vapor phase have seen laboratory and commercial use in the past. Copper brazing of ferrous parts in a reducing atmosphere containing hydrogen and/or carbon monoxide is an established art. Here the reducing gases react with the metal oxides to reduce them to the elemental metallic state and form new gaseous oxides, water and carbon dioxide, leaving no residues. Unfortunately, the utility of this process is limited to a few metals because of the absence from present technology of a stable gas with high reducing power. Thus, hydrogen and carbon monoxide atmospheres are ineffective as fluxes for use with the silver solders and brazing brasses because of their very limited ability to reduce zinc oxide.

Self-fluxing brazing alloys containing phosphorus are applicable to many nonferrous joining problems, reacting with metal oxides to form volatile oxides of phosphorus, and leaving no obnoxious residues. These alloys are, however, not applicable to the brazing of ferrous metals. Ethyl and methyl borate vapors have been successfully applied to fluxing for torch brazing. However, these compounds decompose at elevated temperatures to form boric oxide, which serves as the active fluxing agent in the conventional fashion, limiting the use of this technique to temperatures above that at which boric oxide is fluid, and failing to provide freedom from contamination of the brazed assembly. All of these techniques, then, are of very real but somewhat limited utility.

A single other type of vapor-phase fluxing is known to the authors. Ferrous parts have been pickled for the removal of oxides by heating them in an atmosphere of hydrogen chloride, which reacts with iron oxide to form water vapor and gaseous or liquid iron chloride. This reaction is, however, not unaccompanied by undesired side re-

actions. It is possible that in a completely anhydrous atmosphere, hydrogen chloride would not seriously attack clean steel, but in commercial practice HCl reacts rapidly with the steel surfaces so as to produce undesirable etching effects. Hydrogen chloride has been used as a fluxing atmosphere above cadmium and zinc dipping tanks, but there is reason to question whether fluxing of the steel to be dipped is achieved in the gas phase or through the action of molten chlorides floating on the metal bath. Tests of hydrogen chloride as a fluxing atmosphere for brazing and soldering steel revealed very heavy attack of the gas on steel and



Early laboratory gas fluxing set-up using boron fluoride as a flux.

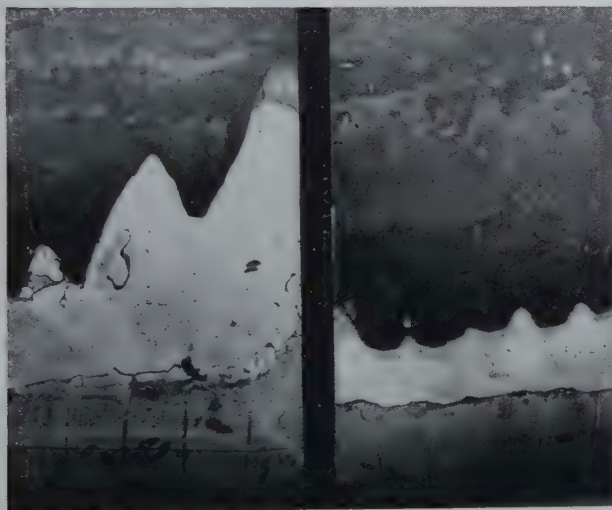
substantial contamination of assemblies with chlorides of one or more of the metals present in the brazing alloy.

Tests were next conducted to determine the feasibility of employing hydrogen fluoride as a gaseous flux for joining steel members by means of brazing alloys. Although this atmosphere produced some etching of steel when employed at temperatures up to 2200°F, surface damage to the steel did not appear severe and good brazing performance was secured with a variety of silver solders and brazing alloys. Careful examination of specimens so prepared revealed no evidence of flux residues in the brazed assemblies. Continued tests of this material fully established the feasibility of employing hydrogen fluoride over a wide concentration range in an otherwise inert atmosphere as a flux for brazing steel. Although no contaminants were found in assemblies fluxed with hydrogen fluoride, some deposition of metal fluorides in the exhaust line from the heated chamber was observed.

Because hydrogen fluoride was regarded as an unattractive material for use in the production shop, efforts were next directed to investigation of other fluoride compounds more readily handled. It was found possible to produce results comparable to those achieved by means of hydrogen fluoride

through the introduction of ammonium fluorides into the brazing chamber in an inert atmosphere. Ammonium fluoride, bifluoride, and polyfluorides were found relatively simple to handle and quite effective as fluxing agents over a wide range of concentration and brazing temperature with a variety of brazing alloys. One difficulty which was encountered in the use of this type of material lay in condensation of sublimed salts in the exhaust lines from the brazing chamber. The lines occasionally became clogged from such condensation and special precautions were required to prevent excessive pressure increase in the brazing chamber. However, as was the case with hydrogen fluoride, satisfactorily brazed assemblies showing no evidence of flux residues were produced by use of ammonium fluorides, purging the system with an inert gas before cooling from the brazing temperature.

In an effort to provide a yet more effective flux, further tests were instituted using ammonium fluoborate as a fluxing agent in an inert atmosphere. This reagent provided fluxing activity su-



Partially torn joint brazed with boron fluoride as a flux. Note that the 4340 steel, not the joint, has failed.

perior to that afforded by hydrogen fluoride or the ammonium fluorides. In use it was subject to the same problems from condensation in the exhaust lines which had characterized other ammonium compounds. However, in all instances it was found possible to prevent such condensation during the early portion of the brazing cycle by passing the exhaust gas from the chamber through a cracking unit heated to a temperature sufficiently high to decompose the ammonium salts and prevent their recondensation. It is indicated that the ammonium fluorides decompose into nitrogen, hydrogen, and hydrogen fluoride and that ammonium fluoborate decomposes into these materials and boron fluoride. In the course of this work it was noted that care must be exercised to secure salts of satisfactory purity. Some samples tested exhibited remarkable fluxing power, while others

were completely inefficient and in some instances even produced scale on the steel surfaces at the lower brazing temperatures.

Boron trifluoride—Because of the excellent fluxing characteristics displayed by ammonium fluoborate, it was next desired to test the fluxing performance of a compound higher in boron concentration. Boron trifluoride appeared particularly attractive in this regard and possessed several other obvious attractive features. It is materially less toxic than the hydrogen fluoride of the other compounds described above, is available as a compressed gas in high pressure cylinders, and is therefore free from a tendency to clog exhaust lines from the brazing atmosphere chamber. It creates a dense white fume on escape into the air, clearly revealing any leaks which may be present in the system. Tests of this material as a flux for brazing of steel yielded most gratifying results. Wetting and flow of molten brazing alloys was superior to that normally exhibited by conventional fluxes used in conjunction with inert atmospheres. No evidence of attack of the atmosphere on steel surfaces could be found and critical spectographic and diffraction studies of steel surfaces brazed in this atmosphere revealed no evidence of contamination. This indicated freedom from surface contamination was reflected in exceptionally good corrosion resistance of the steel surfaces on indoor exposure. As with the other gaseous fluxes studied, the quality of the brazed joint was exceptional and the brazing alloy showed very low incidence of voids.

As was the case with the ammonium compounds tested, the results obtained in brazing steel with a fluxing gaseous atmosphere of boron fluoride indicated considerable variation in performance depending on the concentration and nature of the impurities in the fluxing reagent. This was particularly reflected in the lower temperature limit at which effective fluxing action was provided.

Although a wide variety of other gaseous fluxes have been studied and evaluated, economic and technical factors resulted in the adoption of boron fluoride as the standard flux for this application. Production operations employing this material have been in routine use for a number of months with very gratifying results. Flux removal from assemblies is no longer a problem and brazing quality is excellent.

An effort has been made here to present only in rough outline the application of gaseous fluxing to the brazing of steel. It is desired to emphasize that this process is not advanced as a cure-all and that for a majority of fluxing problems conventional procedures are equal or superior to fluxing in the vapor phase. Gaseous fluxes are relatively slow in action and their fluxing capacity is limited because of their low concentration. They are extremely sensitive to contamination and are often difficult to procure or produce in satisfactory purity. They are generally more costly to buy and to use than are conventional fluxes. However, for critical applications in which conventional fluxing techniques are not applicable, gaseous fluxes may prove invaluable.

Metals Division Fall Meeting

CLEVELAND, OCTOBER 17-19

IN conjunction with the Thirty-First Metal Congress, the Institute of Metals Div., AIME, has scheduled over 50 technical papers for its regular Fall meeting. All sessions will be at the Allerton Hotel, Cleveland, starting Monday morning, Oct. 17 and ending Wednesday afternoon, Oct. 19. The local committee on arrangements is headed by M. D. Harbaugh, vice president and secretary, Lake Superior Iron Ore Association, assisted by Milton Tilley and Reidar Ericksen of National Malleable and Steel Castings Co., and H. Y. Hunsicker, of Aluminum Co. of America.

Registration: Prior to requesting a referendum by the membership on an increase in dues for a three-year period, the AIME Board of Directors voted that all meetings except local section meetings should be as nearly self supporting as possible. Such action was taken since it would be unfair to request AIME members unable to attend meetings for an increase in dues to subsidize meetings. The Metal Congress contributes \$500 each year toward the cost of the Institute of Metals Division's Fall meeting held as part of the Metal Congress. In order to cover the balance of the expenses of the meeting, the Executive Committee of the Institute of Metals Division voted to establish registration fees at the Fall meeting of \$2.00 to members and \$4.00 to non-members. There will be no registration fee for Student Associates of AIME nor for those attending the dinner only. Registration facilities will be available from 8:30 a.m. to 5:00 p.m. at the Allerton Hotel on Monday, Tuesday and Wednesday Oct. 17-19 on the Mezzanine Floor. A package of reprints containing all of the fall meeting papers will be given each registrant at the time of registration.

Annual Fall Dinner: The annual Fall dinner will be held in the Ballroom of the Allerton Hotel

on Tuesday, Oct. 18. At 6:00 p.m., preceding the dinner, there will be a cocktail party in the Chester Room, given with the compliments of the Cleveland members of AIME and their friends and arranged by the Cleveland Local Committee. Tickets for the dinner will admit the holders to the cocktail party. Ladies are cordially invited to the cocktail party and dinner. Dress will be informal.

Institute of Metals Division Fall Dinner

Tuesday, Oct. 18, Allerton Hotel

6:00 p.m.—Cocktail party, Chester Room, for dinner guests, Compliments of the Cleveland Chapter AIME and their friends.

7:00 p.m.—Dinner, Ballroom

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IMD Executive Committee Meeting

Tuesday, Oct. 18, Allerton Hotel

12:30 p.m.—Luncheon, Parlor C

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Powder Metallurgy Committee Meeting

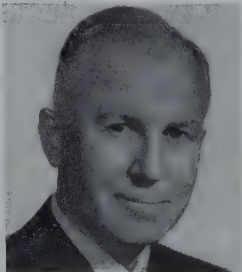
Time and Place to be announced.

* * *

Editorial Advisory Committee Meeting

Wednesday, Oct. 19, Allerton Hotel

12:30 p.m.—Luncheon, Parlor C



M. D. Harbaugh

As in the past, there will be no formal seating plan, although the local committee has made arrangements to reserve complete tables of eight. Dinner tickets will cost \$5.00 each and tables should be reserved by sending a check for \$40.00 per table to Mr. Milton Tilley, National Malleable and Steel Castings Co., 10600 Quincy Ave., Cleveland 6, (Not to Institute headquarters) as soon as possible. Individual tickets will be on sale at the registration desk at the Allerton Hotel during the meeting, and registrants are urged to buy their tickets early when they register in order to enable the local committee to plan the best party possible.

Monday Morning, Oct. 17 — Simultaneous Sessions

BALLROOM

Precipitation and Aging

- 9:00 a.m. to 9:40 a.m. **Precipitation Phenomena in the Solid Solution of Nitrogen and Carbon in Alpha Iron Below the Eutectoid Temperature**, by L. J. Dijkstra, Inst. Study of Metals, U. Chicago, J. of M., March.
- 9:45 a.m. to 10:25 a.m. **Yielding and Strain-Aging of Nitrided Single Crystals of Iron**, by H. Schwartzbart, Res. Met., National Advisory Comm. for Aeronautics, and J. R. Low, Jr., Res. Assoc., G.E.; J. of M., Sept.
- 10:30 a.m. to 11:10 a.m. **Effect of Quenching on the Age Hardening of Two Aluminum Alloys**, by R. D. Barer, Inst. Univ. of British Columbia, and M. B. Bever, Assoc. Prof., M.I.T. J. of M., Aug.
- 11:15 a.m. to 11:55 a.m. **Size Effects in Quenching High Purity Precipitation-Hardenable Alloys**, by W. L. Finlay, Supvr., Remington Arms Co., Inc., Journal of Metals, October.

MATHER ROOM

Fracture and Strain Phenomena

- Metallographic Description of Fracture in Impact Specimens of Structural Steel**, by M. Baeyertz, Sr. Met., E. S. Bumps, Assoc. Met.; W. F. Craig, Jr., Supervisor, Armour Research Foundation. Journal of Metals, August.
- Effect of Ferrite Grain Size on Notch Toughness**, by J. M. Hodge, Res. Assoc., R. D. Manning, Supervising Technologist, and H. M. Reichhold, Asst. Technologist, Res. Lab, Carnegie-Illinois Steel Corp. Journal of Metals, March.
- Investigation of Temper Brittleness in Low Alloy Steels**, by S. A. Herres, Met., and A. R. Elsea, Met., Battelle Memorial Inst. Journal of Metals, June.
- Effects of Molybdenum and Commercial Ranges of Phosphorus on the Toughness of 0.4 Pct. Carbon-Chromium Steels**, by M. Baeyertz, Sr., Met., J. P. Sheehan, Res. Met.; W. F. Craig, Jr., Supervisor, Armour Research Foundation. Journal of Metals, August.

CHESTER ROOM

Recrystallization

- Secondary Recrystallization in Copper**, by M. L. Kronberg, Hammond Lab., Yale Univ., and F. H. Wilson, Copper Res. Lab., American Brass Co. Journal of Metals, August.
- Recovery and Recrystallization in Brass**, by B. L. Averbach, Asst. Prof., Mass. Inst. of Tech. Journal of Metals, August.
- Recrystallization Texture and Coarsening Texture in High Purity Aluminum**, by P. A. Beck, Professor and Hsun Hu, Met. Dept., Univ. of Notre Dame. Journal of Metals, September.
- Recrystallization and Microstructure of Aluminum Killed Deep Drawing Steel**, by R. L. Rickett, Asst. Supvr., Res. Lab., U. S. Steel Corp.; S. H. Kalin, Metallurgist, Carnegie-Illinois Steel Corp., and J. T. MacKenzie, Jr., Sales Engineer, U. S. Steel Export Co., Lisbon, Portugal. J. of M., March.

Monday Afternoon, Oct. 17 — Simultaneous Sessions

BALLROOM

Equilibrium Diagrams

- 2:00 p.m. to 2:40 p.m. **Beryllium-Iron System**, by R. J. Teitel, Assoc. Met., Brookhaven Natl. Labs., and M. Cohen, Prof., Mass. Inst. of Tech. J. of M., April.
- 2:45 p.m. to 3:25 p.m. **Ternary System, Copper - Manganese - Zinc**, by T. R. Graham, Sr. Met.; J. R. Long, Sr. Met., C. E. Armantrout, Met., and A. H. Roberson, Met., U.S.B. of Mines. J. of M., Oct.
- 3:00 p.m. to 4:10 p.m. **Solubility of Titanium in Liquid Magnesium**, by K. T. Aust, and L. M. Pidgeon, Head, Dept. of Met. Engrg., Univ. of Toronto. Journal of Metals, Sept.
- 4:15 p.m. to 4:55 p.m. **Liquid Solubility of Manganese in a Magnesium-Aluminum-Tin Alloy**, by B. J. Nelson, Res. Met., and G. F. Sager, Res. Met., Alcoa, J. of M., July.

MATHER ROOM

Fracture and Strain Phenomena

- Discontinuous Crack Propagation — Further Studies**, by L. D. Jaffe, Met., E. L. Reed, Met., and H. C. Mann, Materials Eng., Watertown Arsenal Laboratories. Journal of Metals, October.
- Stress and Strain States in Elliptical Bulges**, by C. C. Chow, A. W. Dana, Res. Lab. for Mech. Met., and G. Sachs, Formerly Prof. of Physical Met., Case Inst of Tech. Journal of Metals, January.
- Comparative Creep Properties of Several Types of Commercial Coppers**, by A. D. Schwoppe, Res. Engr., K. F. Smith, Res. Engr., and L. R. Jackson, Supvr., Battelle Memorial Institute. Journal of Metals, July.
- Active Slip Systems in the Simple Axial Extension of a Single Crystalline Alpha Brass**, by R. C. Maddin, Asst. Prof., The Johns Hopkins Univ., C. H. Mathewson, Prof., and W. R. Hibbard, Jr., Asst. Prof., Yale U., J. of M., Aug.

CHESTER ROOM

Electrical Properties of Alloys

- P-Type and N-Type Silicon and the Formation of the Photovoltaic Barrier in Silicon Ingots**, by J. H. Scaff, Met., H. C. Theurer, Met., and E. E. Schumacher, Chief Met., Bell Telephone Labs., J. of M., June.
- Microstructure of Silicon Ingots**, by W. G. Pfann, Met., and J. H. Scaff, Met., Bell Telephone Labs. Journal of Metals, June.
- Electrical Properties of the Intermetallic Compounds Mg_2S_n and Mg_2Pb** , by W. D. Robertson, Res. Assoc., Inst. for Study of Metals, Univ. of Chicago, and H. H. Uhlig, Assoc. Prof., Charge of Corrosion Lab., M.I.T. Metals Tech., Oct. 1948.
- Ferromagnetic Alloys in the Systems Copper-Manganese-Indium, and Copper-Manganese-Gallium**, by F. A. Hames, Asst. Prof., Univ. of British Columbia, and D. S. Eppelsheimer, Prof., Missouri School of Mines & Met. J. of M., August.

Tuesday Morning, Oct. 18 — Simultaneous Sessions

BALLROOM

Transformations

- 9:00 a.m. Kinetics of the Austenite Martensite Transformation, by J. C. Fisher, Res. Assoc., J. H. Hollomon, Phys. Met., and D. Turnbull, Res. Assoc., General Electric Co. J. of M., Oct.
- 9:40 a.m. Free Energy Changes Accompanying the Martensite Transformation in Steels, by J. C. Fisher, Res. Assoc., G.E. Co. J. of M., Oct.
- 9:45 a.m. Hardenability Effect of Molybdenum, by J. M. Hodge, Res. Assoc., J. L. Giove, Supervising Technologist, and R. G. Storm, Asst. Technologist, Res. Lab., Carnegie-Illinois Steel Corp. J. of M., March.
- 10:25 a.m.
- 10:30 a.m.
- 11:10 a.m.
- 11:15 a.m. to 11:55 a.m.

MATHER ROOM

Fracture and Strain Phenomena

- Simultaneous Aging and Deformation in Metals, by J. D. Lubahn, Res. Labs., General Electric Co. Journal of Metals, October.
- Transverse Bending of Single Crystals of Aluminum, by M. K. Yen, Res. Assoc. School of Engrg., N. Y. Univ., and W. R. Hibbard, Jr., Asst. Prof. of Met., Yale University. Journal of Metals, Oct.
- Stages in the Deformation of Monel Metal as Shown by Polarized Light, by D. H. Woodard, General Physical Scientist, Natl. Bureau of Standards. Journal of Metals, October.
- Influence of Temperature on the Stress-Strain Energy Relationship for Copper and Nickel-Copper Alloys, by D. J. McAdam, Jr., Former Ch. of Sect., Natl. Bureau of Standards. J. of M., Oct.

CHESTER ROOM

Surface Orientation-Phase Relationships

- Surface Orientation and Rolling of Magnesium Sheet, by R. L. Dietrich, Dow Chemical Co. Journal of Metals, September.
- Oriented Arrangements of Thin Aluminum Films on Ionic Substrates, by Thor Rhodin, Jr., Res. Assoc., University of Chicago. Journal of Metals, June.
- Kinetics of the Reactions of Zirconium with O_2 , N_2 , and H_2 , by E. A. Gulbransen, Advisory Engr., and K. Andrew, Westinghouse Research Laboratory. Journal of Metals, August.
- Kinetics of the Reactions of Titanium with O_2 , N_2 , and H_2 , by E. A. Gulbransen, Advisory Engr., and K. Andrew, Westinghouse Research Laboratory. Journal of Metals, October.

Tuesday Afternoon, Oct. 18 — Simultaneous Sessions

BALLROOM

Transformations

- 2:00 p.m. Stable Transformation to Nuclei in Solids, by J. N. Hobstetter, Asst. Prof. of Engrg. Science, Harvard Univ. Metals Tech., Oct. 1948.
- 2:40 p.m.
- 2:45 p.m. Transformation in β -CuAl Alloys, by E. P. Klier, Asst. Prof., Penn. State College, and S. M. Grymko, Res. Engr., Battelle Memorial Inst. J. of M., Sept.
- 3:25 p.m.
- 3:30 p.m. Transformation of Gamma to Alpha Manganese, by E. V. Potter, H. C. Lukens, Physicists, and R. W. Huber, Physical Met., U. S. Bureau of Mines. Journal of Metals, July.
- 4:10 p.m.
- 4:15 p.m. to 4:55 p.m.

MATHER ROOM

Structure and Identity of Materials

- Rapid Determination of Orientation of Cubic Crystals, by C. G. Dunn, Res. Physicist, and W. W. Martin, Met., Univ. of Calif. Journal of Metals, July.
- Crystal Structure of Ni_3W , by E. Epremian, Res. Lab., and D. Harkner, Res. Assoc., General Electric Co. Journal of Metals, April.
- Structure of Diborides of Titanium, Zirconium, Columbium, Tantalum, and Vanadium, by J. T. Norton, Prof., Mass. Inst. of Tech., H. Blumenthal, Chief Chemist, and S. J. Sindeband, Technical Director, American Electro Metal Corp. Journal of Metals, October.
- Isolation of Carbides from High-Speed Steels, by D. J. Blickwede, Head, High Temp. Alloys Group, Naval Research Lab., and M. Cohen, Prof., Mass. Inst. of Tech. Journal of Metals, September.

CHESTER ROOM

Diffusion—Grain Boundaries

- Diffusion and Solubility of Carbon in Alpha Iron, by J. K. Stanley, Magnetic Dept., Westinghouse Electric Corp. Journal of Metals, October.
- Analysis of Interstitial Diffusion Using Activity Methods, by G. Guy, Assoc. Prof., North Carolina State College. Journal of Metals, September.
- Study of Grain Boundaries with the Electron Microscope, by J. F. Radavish, Dept. of Physics, Purdue University. Journal of Metals, July.
- Studies of Interface Energies in Some Aluminum and Copper Alloys, by K. K. Ikeuye and C. S. Smith, Director, Inst. for Study of Metals, University of Chicago. Journal of Metals, October.

Wednesday Afternoon, Oct. 19 — Simultaneous Sessions

BALLROOM

Annealing Twins—Kink Bands

2:00 p.m. **Origin of Annealing Twins in Brass**, by R. C. Maddin, Asst. Prof., The Johns Hopkins Univ., C. H. Mathewson, Prof., and W. R. Hibbard, Jr., Asst. Prof., Yale University. *Journal of Metals*, September.

2:35 p.m. **Annealing Twins in Copper and 70-30 Alpha Brass**, by W. R. Hibbard, Jr., Asst. Prof., You Chao Liu, and S. F. Reiter, Grad. Students, Yale University. *Journal of Metals*, September.

3:10 p.m. **Structure and Nature of Kink Bands in Zinc**, by J. B. Hess and C. S. Barrett, Inst. for Study of Metals, Univ. of Chicago. *Journal of Metals*, September.

3:50 p.m. **Vapor Pressure of Zinc and Cadmium Over Some of their Silver Alloys**, by C. E. Birchenall, Staff Member, and C. H. Cheng, Res. Asst., Carnegie Inst. of Technology. *Journal of Metals*, July.

4:25 p.m. to 5:00 p.m.

MATHER ROOM

Powder Metallurgy

Method of Examination of Sections of Fine Metal Powder Particles with the Electron Microscope, by Laurence Delisle (Miss), Jr. Engr., Sylvania Electric Prods., Inc. *Journal of Metals*, March.

Pore Size of Hydrogen Reduced Tungsten Powder, by Bernard Kopelman, Section Head, and C. C. Gregg, Sr. Engr., Sylvania Elec. Prods., Inc. *Metals Technology*, August, 1948.

Determination of Boundary Stresses During the Compression of Cylindrical Powder Compacts, by M. E. Shank, Instr., and John Wulff, Prof., Mass. Inst. of Tech. *Journal of Metals*, September.

Dilatometric Study of the Sintering of Metal Powder Compacts, by Pol Duwez, Assoc. Prof., and Howard Martens, Res. Engr., Calif. Inst. of Tech. *Journal of Metals*, September.

Electrical Resistivity Measurements on Iron-Silicon Compacts Prepared by the Powder Metallurgy Procedure, by F. W. Glaser, Res. Engr., American Electro Metal Corp. *Journal of Metals*, August.

CHESTER ROOM

Titanium—Beryllium

Lattice Parameters of High Purity Alpha Titanium and the Effect of Oxygen and Nitrogen on Them, by H. T. Clark, Jr., Res. Met., Remington Arms Co., Inc. *Journal of Metals*, September.

Effect of Oxygen, Nitrogen and Hydrogen on Iodide Refined Titanium, by R. I. Jaffee and I. E. Campbell, Battelle Memorial Institute. *Journal of Metals*, September.

Preparation and Casting of Beryllium Melts, by J. K. Kura, Asst. Supvr., J. H. Jackson, Supvr., M. C. Udy, Res. Engr., and L. W. Eastwood, Supvr., Battelle Memorial Institute. *Journal of Metals*, October.

Metallographic Examination of Beryllium Alloys, by M. C. Udy, Res. Engr., G. K. Manning, Supvr., and L. W. Eastwood, Supvr., Battelle Memorial Institute. *Journal of Metals*, October.

Weirton Builds World's Largest Openhearth

THE Weirton Steel Co., Weirton, W. Va., is at present building a 550-ton openhearth furnace which will probably tap well over 600 tons per heat. This new furnace, by far the largest in the world, is only rivaled by the 500-ton Great Lakes furnace which was initially fired last week. The Weirton furnace is being constructed on the site of a former 200-ton furnace. On May 31, 1949, the last

heat was taken from the 200-ton furnace and it was shortly thereafter dismantled. This included tearing the furnace down completely, including checker chambers, flues, stack and all pads.

The new 550-ton furnace should be ready to light about Sept. 4 and it is expected that the first heat will be tapped about Sept. 10.

The new furnace will be all basic except the main roof and will have

a 100 pct rammed bottom. There will be a single uptake, a slag pocket and checker chamber at each end. The checker roofs will be flat suspended. The furnace control will be of the Morgan-Isley type (2 stacks) and there will be auxiliary checkers in the Isley stacks. The furnace is being designed to fire either oil or tar and the charge will consist of 70 pct blown metal and 30 pct scrap.

Scientists Discuss Powder Metallurgy

The physics of powdered metals was the subject of a symposium arranged by Walter E. Kingston of the Metallurgical Laboratories of Sylvania Electric Products, Inc., Bayside, N. Y. on Aug. 24, 25 and 26. Among the outstanding authorities who



participated and are shown here, left to right, were Dr. Morris Cohen of the Massachusetts Institute of Technology; Dr. G. F. Huettig of the University of Graz, Austria; Walter E. Kingston, chairman of the symposium and manager of Sylvania's metallurgical laboratories; Dr. Adolf Smekal of the University of Darmstadt, Germany; and Dr. A. J. Shaler of the Massachusetts Institute of Technology. More than 125 attended the three day sessions which were held in the Post Theatre, Fort Totten at Bayside.

Refractories Practices Described

The General Refractories Co., Philadelphia, has just published a handbook of complete, accurate and up-to-date information on refractories. It is intended to serve those who design, use, purchase, construct and supervise the operation of equipment requiring refractory materials.

Its 272 pages, attractively and durably bound, cover the subject of refractories more completely

than has heretofore been attempted. Beginning with a brief but interesting history of refractories from the sun-dried brick of old Egypt to the modern all-basic openhearth steel furnace, the book takes up every phase of refractory practice.

It describes the manufacture of refractory brick from raw material selection to the finished product. It tells what industries use refractories, where and why, and provides a guide to selection of the refractories best suited for each use. Grefco's complete line of refractory brick, mortars, plastics and castables is described.

A section of the book lists, pictures, and gives dimensions of all standard refractory brick sizes and shapes. Another deals with the properties and behavior of refractories.

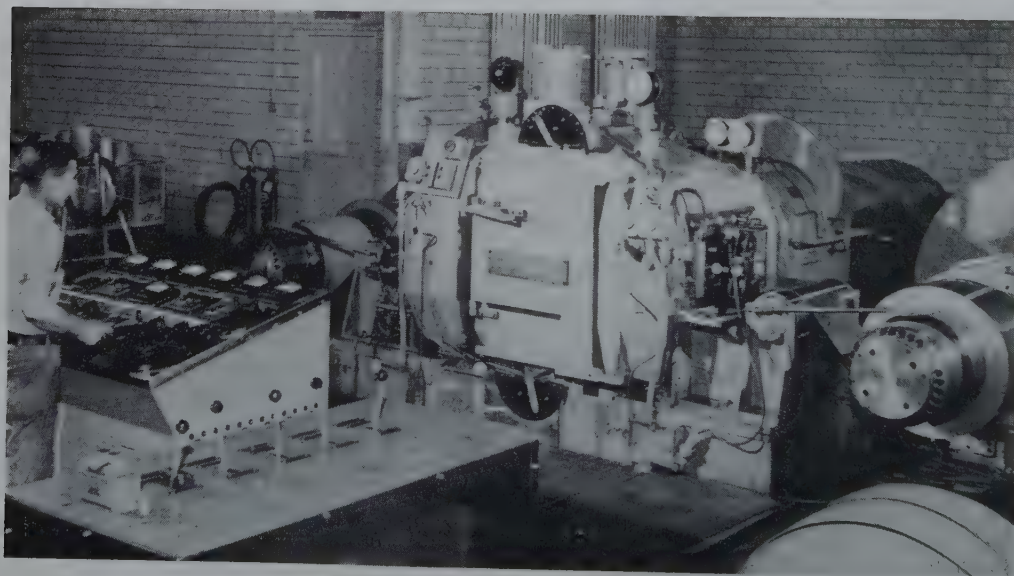
For engineers and architects who design and build equipment that requires refractories there is a great deal of information on construction with refractories as well as tables that simplify the calculation of numbers and types of brick needed.

For the purchasing agent there are the trade practices and other information concerning ordering and shipping of refractory materials.

Perhaps as important as any other section of this refractories handbook is the glossary which defines, in some cases for the first time in print, the meanings of many terms used in this industry. And a complete index at the back facilitates reference to any desired information.

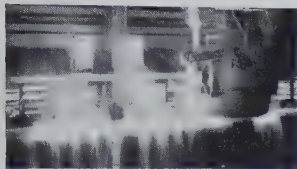
High Temperature Metallic Compound

A new metallic compound which will withstand the high temperatures of modern gas turbines, jet and rocket engines, has been developed under an Office of Naval Research contract with the American Metals Corp., Yonkers, N. Y. The compounding process, using zirconium and boron, provides an alloy which, in recent tests, survived the highest temperature blasts of any materials extensively tested to date.



Republic Steel Corp.'s new Sendzimir mill for cold rolling stainless up to 25 in. wide. This 20-high mill rolls strip down to 0.005 in., but the bulk of tonnage has so far been in the 0.015-in. range.

Open Hearth Meeting, Columbus, Sept. 30



Following the All-Institute midyear meeting of the AIME in Columbus, the National Open Hearth Committee, Southern Ohio Section, will close a busy week with a one-day technical session followed by a Saturday program featuring the Ohio State-Indiana football game. Technical sessions will be held at the Deshler-Wallick Hotel on Friday, Sept. 30.

The committee on arrangements is made up of Robert Bower, chairman, and Vernon Jones, R. W. Lewry and P. G. Sammett. The program follows:

THURSDAY, SEPT. 29

9:00 a.m.—Meeting Program Committee, National Open Hearth Committee.

FRIDAY, SEPT. 30

9:00 a.m.—Registration outside Parlor I.

10:00 a.m.—Opening remarks by Robert S. Bower, Section Chairman.

Operating Meeting: R. W. Lewry, chairman; P. G. Sammett, co-chairman.

- (1) Safety in the Open Hearth, by Elmer Barker, Armco Steel Corp.
- (2) Human Relations in Open Hearth Operation.
 - a—Training.
 - b—Education.
 - c—Moral.
- (3) Economics of the Use of Oxygen, a round table discussion.
- (4) Use of Basic Refractories in England and Sweden, by W. S. Debenham, Carnegie-Illinois Steel Corp.
- (5) The Blast Furnace and Its Effect on the Open Hearth, by A. W. Thornton, National Tube Co.

1:00-2:00 p.m.—Luncheon, Main Ball Room.

2:00 p.m.—Practice and Quality: C. E. Sims, chairman; R. Thornbery, co-chairman.

- (1) Conservation of Manganese.
 - a—Use of Alloys.
 - b—Discussion.
- (2) Vertical Solidification in Steel Ingots, by J. W. Spretnak, Ohio State University.
- (3) Relation of Height of Mold to Quality of Rimmed Steel, a round table discussion.
- (4) Mold Coatings, by J. F. Pollack, J. & L. Steel Corp.
- (5) Effect of Soaking Pit Practice on Surface, a round table discussion.

7:00 p.m.—Dinner Meeting, Main Ball Room.

Toastmaster: C. D. King, Chairman Operating Committees, United States Steel Corp.

Speaker: Henry Pildner, Music Director, Radio Station WGAR, Cleveland.

SATURDAY, OCT. 1

9:00 a.m.—Meeting National Open Hearth Committee.

10:00 a.m.—Plant Visitations.

2:00 p.m.—Ohio State vs. Indiana—Football.

— New Books —

Principles of Metallographic Laboratory Practice, by George L. Kehl, 520 pp., 1949. McGraw-Hill Book Co., \$5.50. It is a pleasure to read a third edition that is even better than the first. This book has been thoroughly rewritten so there is a smooth integration of the old and new material.

As before, the book is confined to a complete and logical presentation of the basic principles of metallographic techniques. Preparation and etching of specimens, metallurgical microscopes, photomicrography, photography, macroscopic examination, hardness testing, determination of grain size and hardenability, some nondestructive methods such as magnetic and supersonic tests, pyrometry and thermal analysis are covered in detail. The author has wisely refrained from outlining laboratory experiments as these would, to a large extent, depend on the available equipment and time. Although the book is primarily intended for the student, many an older metallurgist will find it a profitable reference source for information.

Particular credit is deserved for the appendix, a collection of 61 tables, which is practically a handbook for the metallographist.

Engineering Societies Employment Service

THE following employment items are made available to AIME members on a nonprofit basis by the Engineering Societies Personnel Service, Inc., operating in cooperation with the Four Founder Societies. Local offices of the Personnel Service are at 8 W. 40th St., **New York 18**; 100 Farnsworth Ave., **Detroit**; 57 Post St., **San Francisco**; 84 E. Randolph St., **Chicago 1**. Applicants should address all mail to the proper key numbers in care of the New York office, and include 6¢ in stamps for forwarding and returning application. The applicant agrees, if placed in a position by means of the Service to pay the placement fee listed by the Service. AIME members may secure a weekly bulletin of positions available for \$3.50 a quarter or \$12 a year.

POSITIONS OPEN

Chemist, M.S. or B.S. degree, with three or four years' experience, and thorough knowledge of analytical investigation of metals, high-temperature alloys, production methods and processes. Knowledge of instrumentation, including polarographic and potentiometric apparatus. Will perform variety of standard chemical tests and analyses in laboratory for quality control of such materials as metals and alloys, petroleum products, paints, etc. Will set up procedures for routine testing and supervise tests done by graduate chemists and technicians, and devise apparatus for tests to be performed on a fast-moving production line. Location, Connecticut. Y-1839.

Graduate Metallurgist, preferably with M.S., for full-time work on a project on the Mechanical Properties of Single Crystals of Pure Iron. Opportunity for advanced study in night school. Give full qualifications and salary required in letter. Metals Research Laboratory, Carnegie Institute of Technology, Pittsburgh 13.

Assistant Shop Superintendent with experience in steel fabrication for a shop fabricating structural steel, miscellaneous, and ornamental iron and resistance-welded grating. Location, Tennessee. Y-2331.

Plant Engineer, graduate preferred, with some experience in furnace and mechanical design. Knowledge of metallurgy helpful but not necessary. Will design equipment including furnaces, mechanical devices, simple facilities for the handling of parts in and out of furnaces, etc. Will supervise the construction and maintenance of equipment. Salary dependent upon qualifications. Location, Pennsylvania. Y-2494.

Production Manager-Chemical Engineer. Must have operating knowledge of equipment and long experience in the treatment of nonferrous ores. Salary and bonus. Write Journal of Metals, Box A-100.

Engineers: (a) Chief Draftsman for engineering room of about 30 men. Should be experienced in supervising men and allocating work, and have experience in design of rolling mills and auxiliary equipment for ferrous and/or nonferrous metals; (b) Assistant to Supervisor of the rolling mill estimating department, must be experienced in layouts of general-type mills for ferrous and/or nonferrous metals and should be capable of estimating weights and writing specifications for proposals; (c) Designers, Layout Men and Draftsmen for rolling mills and auxiliary equipment for ferrous and nonferrous metals. Must have the above experience. Salaries open. Location, New York, N. Y. Y-2457.

Design Engineer, 35 to 40 yr, Bachelor's degree and experience in machine design, preferably on equipment involving the forming and fabricating of metal parts or assemblies. Knowledge of welding procedure helpful. Company specializes in the manufacture and sale of flexible metallic hose and fittings. Will be responsible for the details of the solution of engineering problems and the application of the results. Will institute and maintain contacts with manufacturers and vendors of special equipment which might be used for the purpose above. Salary, \$6600-\$7200 a year. Location, Connecticut. Y-2673.

Chief Engineer for tank and plate fabricating plant, capable of designing, estimating and selling this type of work, as well as supervising other employees in the same work. Should have broad experience in following jobs through to completion. Location, South. Y-2638.

Metallurgists, 2, young, to work as shift bosses. Single status. Opportunity for advancement to assayer, etc. Prefer men with some experience, but will consider recent graduates. Two-year contract. Salary \$2400 plus room and board. Location, Central America. Y-2641.

Engineer with some experience on methods-engineering work. Should be able to develop jigs and fixtures for use in the quantity production of metal parts, and should have imagination and initiative. Location, Ohio. Y-2650C.

Sales Engineer to contact the steel, refractory and abrasive industries for the sale of ores. Base salary, plus commission, plus expenses. Location, New York, N. Y. Y-2666.

Assistant Professor, metallurgy. Salary open. Location, Pennsylvania. Y-2686.

Instructors and Research Workers, in metallurgical engineering, recent graduates of good scholastic ability, who desire part-time or full time employment in teaching or in research while studying for an advanced degree. Research opportunities are available in light alloys, welding and powder metallurgy. Location, upstate New York. Y-2708.

Graduate Metallurgist, foundry foreman, 30-45 yr. Three to five years' experience as supervisor of foundry operations, completely informed on all production and job-foundry work from small parts up to 1 ton. Will direct small shop operations for a manufacturer. Salary open. Location, Chicago area. R-5797.

Chemical-Metallurgical Engineer, graduate chemist or metallurgist, young, some experience with laboratory analysis and test inspection of ferrous metals or metal products received from vendors. Will do analysis conforming to specifications—hardness testing, set up laboratory and test. Will set up all testing procedure and equipment, make analysis and tests to assure conformance with specifications. Consult with and advise others in regard to metallurgical problems. Salary open. Location, Iowa. R-5832.

MEN AVAILABLE

Executive Metallurgist, broad manufacturing background in metals and finished products, and overall experience in management, internal problems, reorganization, sales, research, cost and quality control, plant layout and modern production-line technique. Sufficiently experienced for maximum responsibility. Seeks executive or consulting activity. Any location and willing to travel. Available immediately. M-458.

Metallurgist, 29 yr, degree in Met. Engrg. Experienced physical testing, assay work, electrolytic silver-gold production and short period in experimental powder metals laboratory. Desires position in or leading to production work. Midwest or eastern U. S. M-459.

Foundry Metallurgical Engineer, eight years' practical experience all phases of foundry metallurgy and operation. Thoroughly familiar with melting, gating, sand, quality and processes control and the decrease of foundry defects for gray iron and aluminum castings. Extensive experience in physical testing, sand testing, and metallographic laboratory work. College graduate, 31 yr, excellent recommendations. M-460.

Metallurgical Engineer, two degrees, nonferrous experience, sales and purchases. Two years' smelting, production and control. Desires good job with opportunity for use of executive ability. New York City or vicinity. M-466.



aime NEWS

Directors, at Special July Meeting, Amend Bylaws and Appoint Lippert Publication Manager

Ordinarily the Board of Directors of the Institute does not meet in July and August but several matters of importance made such a meeting necessary this year. President Young presided, with eleven other members of the Board present: Messrs. Daveler, Elkins, Head, Kinzel, Kraft, Meyerhoff, Peirce, Phillips, Price, Schumacher, and Fletcher. The date was Aug. 17.

Revisions in the bylaws, as printed in the June issues of the journals, were approved without further discussion. These increase the dues by \$5 for Members and Associate Members, and by \$2 for Junior Members, for the years 1950, 1951, and 1952, at which time another referendum will be taken as to the scale for the future. Also, the grade of Junior Foreign Affiliate is discontinued at the end of the current year, and the time in which a Student Associate may remain such is now limited to the end of the year in which he is in university residence as a student, instead of the end of the following year as before.

Appointment of Thomas W. Lippert as Manager of Publications on a two-year contract was confirmed by the Board, and he was introduced to those present. Comment on this appointment appears in "The Drift of Things."

Considerable discussion developed, inspired by a letter from Charles T. Holland, on whether or not a change should be made in the scale of Student Associate dues. At present, Student Associate affilia-

tion, including a year's subscription to one of the three journals, is \$4.50, but an alternative of \$2 is offered without an individual subscription to a journal, in which case copies are supplied to the Affiliated Student Society for reference on request. A proposal had been made to eliminate the \$2 fee, and possibly to reduce the \$4.50 fee to \$4. About one quarter of the Student Associates currently pay \$2 and three quarters \$4.50. The views of several members of the Mineral Industry Education Division were reported, and the student dues schedules and privileges of the other Founder Societies compared with those of the AIME. Lengthy discussion of the matter at the El Paso meeting in 1948 was reviewed. It was finally unanimously decided to retain the present bylaw as to student dues.

Another matter for discussion was the terms under which AIME members should receive the annual volume, "Statistics of Oil and Gas Development and Production," for 1950. The last two volumes have cost from \$10,000 to \$12,000, which is offset only to the extent of \$5000 by receipts from the Doherty Memorial Fund. Several years back the material therein contained had been supplied to Petroleum Division members bound in with the regular petroleum volume each year and the extra cost of binding it separately was supposed to be met by the Doherty Fund contribution. Mr. Elkins explained the matter to the rest of the Board, which

then voted to make the 1950 volume available free on request to all Members, Associate Members, and Junior Members who take the **Journal of Petroleum Technology**, but to charge Student Associates \$3 should any of them wish to have the volume, and to charge others \$6.

Reports were made of a continuing investigation into possible economies in printing, and Mr. Lippert promised further data at the Sept. 27 Board meeting in Columbus.

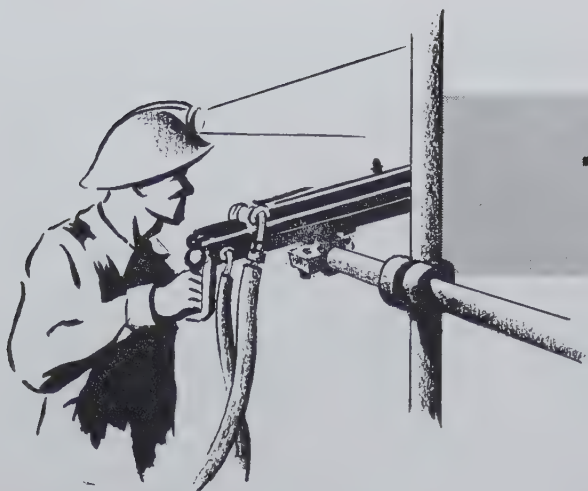
A new Student Chapter was recognized at Fenn College, Cleveland.

H. G. Moulton was named as AIME representative on the Engineers Joint Council Committee on Engineering Personnel in the Military Establishment. The date of the November Board meeting was set at 2 p. m., Wednesday, Nov. 16.

Mr. Peirce made a short progress report of the work of his committee on Instructions to the Nominating Committee, and was directed to expand the work of his committee to include a study of the basic features of the present system of member representation through Directorships.

Currently new members do not receive copies of the journals until they have accepted election, but it was voted to return to the old system of putting applicants on the subscription list for the journal of their choice as soon as their applications are received.

Special messages of congratulation were voted to Herbert Hoover on the occasion of his 75th birthday anniversary on Aug. 10, and to D. C. Jackling, on his 80th birthday on Aug. 14.



THE DRIFT OF THINGS

... as followed by EDWARD H. ROBIE

A Manager of Publications

Among the major recommendations of the Johnson Committee, as reported to the Board at the Annual Meeting last year, was "that the services of an editor and publisher of broad practical experience in this highly technical field should be procured as soon as circumstances permit." This and some other recommendations and suggestions of the Committee have not been forgotten and another step in carrying them out was made in July when T. W. Lippert was engaged as Manager of Publications.

Although the desirability of engaging a man thoroughly conversant with editorial, advertising, and circulation problems in the AIME's field had been recognized, the possibility of securing a qualified man, and especially at a price the Institute could afford to pay, had not been thought too bright. But on June 21 a short news item in the New York newspapers told of a major reorganization of the staff of *The Iron Age*, which had been followed by the resignation of the directing editor, Tom Lippert.

The possibilities for the Institute in this news item were apparent. Tom Lippert had established quite a reputation on *The Iron Age*, not only as an editor but in assisting the "publisher," and the magazine itself has become perhaps the most successful publication in the mineral industries field. Furthermore, Tom had been a member of AIME for fourteen years, joining two years after receiving his M.S. at Carnegie Tech in 1932, so was familiar with Institute affairs. Within a week after the news broke, a tentative agreement had been made between the Secre-

tary and Mr. Lippert for a two-year contract, by the end of which time it was felt that his ability at improving the Institute's journals and increasing their net income could be proved. The whole story was mailed to the 36 Directors of the Institute on July 1, after several had been personally approached or had given their tacit approval by telephone. Twenty-nine of the 36 replied, unanimously approving the proposal, and Lippert's engagement was formally ratified by the Board at its July 27th meeting. Quick action had been necessary because other opportunities awaited him.

Tom's experience has been principally in the field of *The Journal of Metals*, so his immediate duties will be chiefly concerned with the editorial and advertising departments of that journal, to which a major proportion of his salary and expenses will be charged. He will, however, also supervise the editorial and advertising activities of *Mining Engineering*. For the time being, at least, these two journals will take up all of his time, but if and when the Petroleum Branch wishes to avail itself of his services it is hoped that he can extend his interests to that publication. He is also nominally for the present in charge of all Institute book publishing.

Although this step means an immediate increase in AIME expenses, it is confidently expected by all concerned that by a year from now a net gain will result, and that editorially the journals will be even more attractive than at present. It might also be mentioned that the combined salaries of the new Manager of Publications and the Secretary are less than were the com-

bined salaries of the former Secretary and of the Assistant Secretary (who has now become Secretary) so no increase has thus been made in Institute salary expenditures in the "top brass" category.

Naming Committeemen

In naming chairmen of committees, and to some extent in nominating men for Directorships in the Institute as well, one is faced with two alternatives: (1) to name a man for another term, if he has done an exceptionally good job; or (2) to pick an entirely new man. The advantages and disadvantages of each principle are obvious. We are inclined to favor the second alternative, but with this general qualification—that the man selected shall already have served a term as vice-chairman, or at least as a member of the committee, so that he can be reasonably familiar with what he is supposed to do. Certainly we think there is nothing more likely to make for dry rot, nor to annoy those who are interested in a committee's work but never receive recognition, than to retain the same committee chairman and personnel every year, with only an occasional introduction of new blood, perhaps when somebody dies.

One of course likes to retain the services of a man who has done a job with conspicuous success; it perhaps may be regarded as an honor to him, and of benefit to the Institute, to ask him to serve again, but on the other hand, he is probably a busy man who has a salary to earn, and a good and willing horse should not be overworked.

As to the plain committeemen, those who never find their names on a committee sometimes complain that they go entirely unrecognized, and that the group seems to be a little clique of insiders. It has been

our experience, however, that this is not so, but rather that the practice of continuing names on a committee results from the not unnatural human tendency to do a job the easiest way. Many willing and capable men are entirely unknown to the person who picks a committee. Here is where modesty is a fault. If you wish to work on a committee, and feel that you can contribute something to its work, it devolves upon you to let yourself be known. The Executive Committee or the Chairman of your Division will be only too glad to know of your willingness to do committee work. So will the Secretary of the Institute. Don't wait for a formal invitation, or remain aloof in the hope that your name will some time be drawn out of the hat—your name probably isn't in the hat at all.

You haven't time for committee work, anyway, you say? Nonsense. That means you haven't time to work with the men engaged in an activity similar to yours, to find out what they are doing, and what others in your field are doing, to make personal friends who may in a five-minute telephone conversation tell you what it would take you five days of research to find out all by yourself.

If you are not satisfied with what you are getting out of the Institute, the solution of your difficulty may well be connected with the answer to a question that you can ask yourself: What am I **contributing** to the Institute? If you reply that you pay your dues regularly, that is an inadequate answer.

Two Unions Sign Up

Faced with the fact that efforts to repeal the Taft-Hartley Act have been unsuccessful in the present session of Congress, the International Union of Mine, Mill, and Smelter Workers and the United Steel Workers have decided to sign the non-Communist affidavits required under the Act in order to secure recognition as bargaining agents by the National Labor Relations Board. As to the Steel Workers, this may presage action through the NLRB in the current wage-pension dispute with the steel industry, which is enjoying a two-months' truce expiring Sept. 15. As to the IUMMSW, the action seemed necessary in order to protect itself

against the competition of rival unions in the CIO. Heretofore it has not had the right to have its name appear on NLRB ballots. Not only has it been disliked by employers who have had to deal with it, but has also been torn with internal dissension. It is well known, or at least it is currently believed, that the IUMMSW is directed to a considerable extent by Communists and fellow travelers, and whether or not any change in its officers is planned or not remains to be seen. In any case the NLRB and the Department of Justice might well co-operate in a close study of the non-Communist affidavits.

Free College Educations

Another step in making it easy to get a professional education is reported from Argentina where tuition will no longer be charged by the National University of Buenos Aires. All citizens who can pass the entrance examinations have a chance for six years' study at state expense. They can become doctors, architects, engineers, or select any other profession. Six years of study will be provided in the schools and colleges under the supervision of the University, the cost of which heretofore has been about \$2400.

In the United States the nearest similar type of education is that which has been provided by the Government under Public Law 346, the so-called G. I. Bill. Last year close to a million veterans were in schools under the terms of this act, which gives up to four years' training, depending on the duration of their active military service. As much as \$500 a year is allowed for tuition fees, books, and supplies; and from \$75 to 120 a month for subsistence.

State universities provide an education at nominal tuition fees to state residents, and several city universities and colleges give free or nominal tuition to city residents, but we have no Federal university.

Here in New York State a movement is on foot to set up a state university, and we have been approached as to whether or not a mining course should be included. For years there has been something at Albany called the University of the State of New York but it exists in name only so far as the popular understanding of the term goes. Our reply to the ques-

tionnaire we fear was not too encouraging; we do not feel that a new school in New York State to teach any branch of mineral technology is needed.

Private Enterprise

Doubtless it's an old yarn, for it came to us via New Zealand and Canada, and our eyes and ears have not been attuned much in the last year to the lighter side of life, but for the benefit of those to whom it is new, we offer the following anecdote in somewhat abbreviated form:

A foreign reporter was trying to find out how the working class in America felt about our capitalistic practices, and among others interviewed a typical factory workman.

"What do you think about the way the workman fares under the American system?" he asked. "Why, I think it's wonderful." "You do? Why?" "Well, in so many ways. Just an example: You finish a hard day's work and are waiting at a near-by corner for a bus to take you home. Along comes the boss, riding alone in a big car, and he stops and asks if he can give you a lift. He asks where you live and you tell him you live in the city about ten miles away. He says he will be glad to drive you there, that he wants to get your point of view and talk over your work, but must stop at his house first and make an important telephone call. You tell him you'll be glad to wait, and while he is making his call he invites you in for a drink. After that is finished he says dinner is ready so you might as well stay and he will drive you home afterward. After dinner you're sitting around the living room chatting with the boss, when he says, "It's getting pretty late so you might as well stay right here and I'll drive you to work in the morning." This seems like a sensible suggestion, so you agree.

By this time the reporter's eyebrows were raised in amazement at this evidence of American democracy. "Do you mean to say," he asked, "that this sort of thing actually happens under capitalism?" "I certainly do," replied the workman. "But," persisted the inquirer, "did it ever actually happen to you?" "No," admitted the workman, "but it happened to a girl in our plant."

Things to Do

AIME and ASME Hold Joint Fuels Conference at French Lick



E. R. Price, Chairman,
Coal Division, AIME

French Lick, Ind., is a particularly pleasant place in October. Plan to be at the French Lick Springs Hotel on the 26th and 27th. In addition to the usual inducements, the 12th annual joint meeting of the Coal Division, AIME, and the Fuels Division, ASME, will attract hundreds of engineers in the coal and fuel industries.

The opening session will be devoted to a symposium on the dewatering and drying of coal. You'll want to hear V. F. Parry and E. O. Wagner report on their experience in drying fine coal at the Bureau of Mines experiment station at Golden. F. P. Calhoun and E. C. Carris come next with two papers dealing specifically with flash drying. The symposium closes with a paper by J. L. Erisman on Coal Drying in Multi-Louvre Dryer.

Thursday morning will be devoted to a discussion of froth flotation of coarse coal particles by R. E. Zimmerman and S. C. Sun; a description of the laboratory control practiced at the mines of the Ayrshire Collieries by J. J. Merle and R. A. Mullins; and a paper by T. C. Spicer on the use of ignition baffles with single retort stokers.

Thursday afternoon a trip is planned to the Maid Marion strip mine of the Central Indiana Coal Co., where visitors will be able to see some of the latest earth moving machinery and the operation of a coal dryer of R. G. Baughman's design. Mr. Baughman, who is general superintendent of preparation and construction for Central Indiana, is conducting

the trip to the mine, some 35 miles from French Lick.

Start making your plans now to attend the meeting. Registration blanks and further particulars will be sent to members of the two divisions about six weeks before the meeting.

While the principal attraction is presumed to be the technical sessions, experience in the past affords eloquent testimony that those who attend will have a good time on the golf course, comfortable porches, and in smoke-filled rooms.

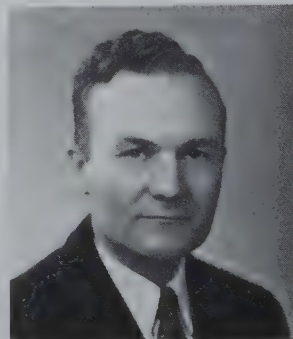


E. R. Kaiser, Chairman,
Coal Div., Program Committee

75th Anniversary at Colorado Mines

Ben Parker, president of the Colorado School of Mines, extends a cordial invitation to members of the AIME to attend the School's 75th anniversary of its founding on Sept. 20 to Oct. 1. Conferences on Friday and Saturday will include coal mining, geophysics, metallurgy, metal mining, geology, and several phases of the petroleum industry.

The Industrial Minerals Division of the AIME, is holding a session in conjunction with the celebration.



Ben Parker, President,
Colorado School of Mines

How to Get the Most From the Meeting

Attending a meeting profitably is the hardest kind of work; calls for a variety of skills; pays good dividends to the successful. The mind must go along with the body.

Every experienced meeting-attender has his own ideas on how to get the most out of the event. Here are a few from an observer's handbook.

Seek new contacts, unfamiliar ideas. A convention is a place where people come together for a purpose. Shun the people and you miss the purpose. To return empty-handed from any gathering is just as wasteful as to return empty-handed from a sales trip.

Neither pure extroverts nor pure introverts get the most out of conventions and meetings. One talks so much he doesn't hear much except the sound of his own voice. The other keeps to himself and hears nothing he couldn't read in some publication next week.

Listening closely to speakers is a small part of

profit-taking from meetings. You must be trying, as well, to analyze, to poke holes in the statements; to translate the ideas into terms of your own problems.

Alert listening breeds questions, stimulates additional ideas. In discussion, ask those questions, express those ideas. A good session at a convention should be a sort of intellectual "Hellzapoppin'."

Make yourself available for committee activity. It often associates you with leaders; makes you important to your own organizations; gives invaluable experience in getting results through group cooperation. When you get back home, finish your convention-attending job. If your boss wasn't smart enough to ask for a memorandum about the meeting, write one anyhow. Fill it with facts, but point the meaning of each fact in relation to your company and your job.

Then you are through attending that meeting... and you will be glad of it! (Excerpts from "Getting Along With Others in Business," by N. G. Shidle.)

Horace M. Albright, president of the U. S. Potash Co., has been elected chairman of the National Minerals Advisory Committee, succeeding Donald H. McLaughlin, president of the Homestake Mining Co., and nominee for AIME President in 1950.

George M. Anderson, former Missouri School of Mines student, can now be reached at the Harbison-Walker Refractories Co., Fulton, Mo.

Robert W. Berkhahn has been employed as a metallurgist by the American Smelting and Refining Co. in El Paso, Texas.



Clyde Williams (left) and Everette De Golyer out for a stroll in Washington. Our reporter notes "at least two ex-Presidents of AIME are able to perambulate."

T. P. (Josh) Billings has become consulting mining engineer for the U. S. Smelting Refining and Mining Co. **Arch G. Kirkland** succeeds him as manager of Western mines. Mr. Billings started his mining career in 1903 at Birmingham where he had charge of Tintic and Bingham operations of the Bingham Mines Co. In January 1929 he joined the U. S. Smelting organization as assistant to the manager of mines. Mr. Billings was given the position of manager of Utah mines in 1934 and in 1936 was advanced to assistant general manager of mines. He has been manager of Western mines since 1943.

Mr. Kirkland is a graduate of Queens University at Kingston, Ont. He was manager of Mic Mac Mines, Ltd., a subsidiary of the U. S. Smelting, for six years previous to his coming to Salt Lake City as assistant manager of western mines in February 1947 which followed the company's sale of the Mic Mac interest. In January 1949 he was made assistant manager of Western mines. Mr. Kirkland had been employed by the Hollinger Gold mines and Hard Rock Gold mines, both in Canada, before his Mic Mac position.

Louis S. Cates expected to go to Africa on July 31 and will be back some time in October.

Nicholas Chlumecky graduated last spring in mining engineering at the University of British Columbia, and is now with the Britannia Mining and Smelting Co., Britannia Beach, B. C.

Norman Cleaveland, formerly of Berkeley, Calif., has gone to Kuala Lumpur, Malaya, as manager of the Pacific Tin Consolidated Corp.

Ward Carithers, who had been working for the Chelan division of the Howe Sound Co. in Holden, Wash., is currently mining geologist for the Calera Mining Co., Forney, Idaho.

Jack Christie has been made assistant underground manager of the N'Kana mine of the Rhokana Corp., Kitwe, N. Rhodesia. He started his mining career at the N'Kana mine fifteen years ago, with unbroken service. Last spring he visited the Witwatersrand gold mines and the Premier diamond mine, studying new mining methods.

John J. Collins, geologist with the U. S. Geological Survey, has taken up residence in Washington, D. C., at 2121 Virginia Ave., N.W. He has been assigned to the mineral resources section of the Survey, his project being an estimation of the resources and exploration possibilities for copper in the United States.

S. G. Lasky is chief of the newly created mineral resources section.

R. P. Connett, formerly with the Fredericktown Lead Co.'s Valle mines in Missouri, is now with the United States Gypsum Co. at Albaster, Mich.

Forbes B. Cronk, after 44 years of continuous service, retired as general mining engineer of the Oliver Iron Mining Co. on July 1. He started work for Oliver as a mining engineer on the Mesabi, earning recognition of his capabilities that

resulted in his becoming chief engineer of the Coleraine district in 1910. After correlating exploration and mining developments, he was transferred to Duluth headquarters as second assistant general mining engineer. His appointment as general mining engineer came in 1945. Mr. Cronk will continue with the Company in an advisory capacity.

H. W. deVriendt, who was a director of the Billiton tin smelter at Arnhem and an adviser of Billiton, has become general director of S. A. Belgochimie, Ghent, Belgium.

W. G. Donaldson has returned from Honduras after developing and equipping the San Andres Mine, a gold property owned by the New Idria Honduras Mining Co. in the western part of the country. The cyanide plant has a capacity of 250 tons per day and went into production last October. Mr. Donaldson now resides at 50 Alta Rd., Berkeley, Calif.

Tell Ertl is now a mining engineer with the Union Oil Co. of California, at Rifle, Colorado.

E. W. Felegy is now an assistant supervising engineer for District F, Safety Branch, Bureau of Mines in Duluth. His home address is 4842 London Rd., Duluth 4, Minn.

William T. Folwell is now mining engineer for the Hecla Mining Co., Burke, Idaho.

Glenn H. Fritz left the States in June for Calcutta, India, where he will represent the Joy Mfg. Co. Mr. Fritz joined the Company last fall, and underwent a period of intensive training in the application of Joy equipment to mining and tunnelling operations. Prior to joining Joy, he had been assistant engineer with Anaconda, training men for surveying and operating work at Butte. Mr. Fritz is no stranger to the Far East, having served with the USAF there during the recent war. Mrs. Fritz accompanied her husband to India, and they can be reached c/o Volkart Bros., Inc., 8 Netaji, Subhas Rd., Calcutta.

Louis Gence has resigned as manager of the Jean Felli Mica Mines, Bekily, Sud Madagascar, and is now connected with the Societe des Minerais de la Grande Ile as mine superintendent of their Ambatoabo mine

about forty miles northwest of Fort Dauphin, Madagascar.

John Griffen, coal preparation engineer with the Pittsburgh office of the McNally Pittsburgh Mfg. Corp., sailed on the Queen Elizabeth on July 15 for a two month inspection tour of European coal cleaning plants. He'll visit with government

and private coal preparation specialists in England, France, Belgium, and the Netherlands.

George C. Heikes, formerly with the Kennecott Copper Corp., has gone to Athens, Greece, with the ECA. His address is ECA, Athens, APO 206, care of Postmaster, New York City.

Samuel B. Kanowitz is eastern district manager of the Raymond pulverizer division of the Combustion Engineering Co., 200 Madison Ave., New York City.

Leon D. Keller resigned as chemist with the Sullivan Mining Co., Kellogg, Idaho, in March to accept a post as metallurgical engineer with The Dorr Co. He is engaged at present in doing heavy chemical and metallurgical research at the Westport mill of the Company.

Sherwin F. Kelly has been made chairman of the Geophysics Committee of the Canadian Institute of Mining and Metallurgy for the coming year.

George A. Kiersch is currently employed as engineering geologist by the Sacramento District, U. S. Corps of Engineers since resigning from the faculty of the Montana School of Mines. Dr. Kiersch spent over a year doing geological research connected with underground construction, and at present is resident geologist, Folsom Dam, where a large exploration program is underway. He resides at 4011 Berkshire Ave., Sacramento 17, Calif.

Paul P. Kraai is working for Bechtel Bros. Construction Co. at South Fork, Calif. His mailing address is Yosemite Forks, Oakhurst. He was working in the department of mining and metallurgy of the Twinning Laboratories at Fresno.

Arnold B. Landstrom, mill superintendent for the Isle Royale Copper Co. until the firm ceased mining operations, has become associated with the research department of the Calumet & Hecla Consolidated Copper Co.

Thomas E. Luebben, research engineer with the Hegeler Zinc Co., Danville, Ill., was formerly with the Chicago-Harrisburg Coal Co.

Louis C. McCabe, former coal branch chief in the Bureau of Mines and for the last two years director of the Los Angeles, Calif., Air Pollution Control District, rejoined the Bureau on July 5 as chief of air and stream pollution prevention research. He will supervise work in this field and will assist the mineral industries in improving methods and devices designed to prevent the emission of harmful substances from mines, mills, smelters, and other installations. He will also serve as assistant chief of the fuels and explosives division under **A. C. Fieldner**, chief of the division. While on the West Coast Dr. McCabe was largely responsible for the progress recently made in developing ways and installing equipment designed to control hazardous air conditions around Los Angeles.

Calendar of Coming Meetings

SEPTEMBER

- 12 Mid-Continent Section, AIME.
- 14 El Paso Metals Section, AIME.
- 15 Carlsbad Potash Section, AIME.
- 15 North Pacific Section, AIME.
- 15 Utah Section, AIME.
- 15-16 Magnesium Association, Hotel Statler, Detroit.
- 16 Oregon Section, AIME.
- 20 Gulf Coast Section, AIME.
- 20 Washington, D. C., Section, AIME.
- 21 Southwest Texas Section, AIME.
- 26 Alaska Section, AIME.
- 27 Montana Section, AIME.
- 29 American Iron and Steel Institute, Hotel Statler, Buffalo.
- 25-28 American Mining Congress, Metal Mining Convention, Hotel Davenport, Spokane, Wash.
- 25-28 Mid-year Meeting, AIME, Neil House, Columbus, Ohio.
- 29-30 ASME, fall meeting, Erie, Pa.
- 29-Oct. 1 Colorado School of Mines, 75th anniversary celebration.
- Sept. 30-Oct. 1 Southern Ohio Section of Open Hearth Committee, AIME, fall meeting, Deshler-Wallick Hotel, Columbus.

OCTOBER

- 3-4 National Assn. of Corrosion Engineers, Adolphus Hotel, Dallas, Texas.
- 3-6 Assn. of Iron and Steel Engineers, William Penn Hotel, Pittsburgh.
- 5-7 Petroleum Branch, AIME, fall meeting, Plaza Hotel, San Antonio, Texas.
- 6 American Iron and Steel Institute, Drake Hotel, Chicago.
- 10-14 American Society for Testing Materials, Fairmont Hotel, San Francisco.
- 13-14 Texas Mid-Continent Oil and Gas Association, annual meeting, Rice Hotel, Houston.
- 14 Eastern Section, Open Hearth Committee, Iron and Steel Division, annual all-day fall meeting, Warwick Hotel, Philadelphia.
- 14 Southwestern Section, Open Hearth Committee, Iron and Steel Division, Kansas City, Mo.
- 17-19 Institute of Metals Division, AIME, fall meeting, Allerton Hotel, Cleveland.
- 17-21 National Metal Congress and National Metal Exposition, Public Auditorium, Cleveland, Ohio.
- 17-21 American Society for Metals, annual meeting, Cleveland, Ohio.
- 17-21 American Welding Society, annual meeting, Cleveland, Ohio.
- 17-23 AIEE, 1949 Mid-West meeting, Netherland Plaza, Cincinnati.
- 19-20 Society for Non-Destructive Testing, Cleveland, Ohio.
- 20-21 Petroleum Branch, AIME, Elks Club, Los Angeles.

24-28 Thirty-seventh National Safety Congress and Exposition, Chicago.

26-27 Joint Fuels Conference, ASME-AIME, French Lick Springs Hotel, French Lick, Ind.

28 Pittsburgh Section of Open Hearth Committee and Pittsburgh Section, AIME, annual fall meeting, William Penn Hotel, Pittsburgh.

28-29 ECPD, annual meeting, Edgewater Beach Hotel, Chicago.

NOVEMBER

- 1-5 Pacific Chemical Exposition, California Section, American Chemical Society, San Francisco Civic Auditorium.
- 2-4 American Society of Civil Engineers, fall meeting, Washington, D. C.
- 7-10 AIChE, annual meeting, Pittsburgh, Pa.
- 7-12 International congress on tunnel driving in rock formation, organized by the Societe de l'Industrie Minerale. Information on meeting available from French Mining Mission, 1322 18th St., N. W., Washington, D. C.
- 9-11 Industrial Minerals Division, AIME, Tampa, Fla.
- 10-14 ASTM, first Pacific area national meeting, San Francisco, Calif.
- 12-14 Geological Society of America, annual meeting, Hotel Cortez, El Paso.
- 14 Arizona Section, AIME, annual meeting, Pioneer Hotel, Tucson, Ariz.
- 16-18 Industrial Hygiene Foundation, 14th annual meeting, Mellon Institute, Pittsburgh.

DECEMBER

- 7 American Mining Congress, Annual Business Meeting, New York City.
- 8-10 Seventh Annual Conference, Electric Furnace Steel Committee, Iron and Steel Division, AIME, Hotel William Penn, Pittsburgh.

JANUARY 1950

- 18-20 American Society of Civil Engineers, annual meeting, New York.
- 30 AIEE, winter meeting, New York.

FEBRUARY 1950

- 10 Southwestern Section, Open Hearth Steel Committee, Iron and Steel Division, St. Louis, Mo.
- 12-16 Annual Meeting, AIME, Statler (Pennsylvania) Hotel, New York City.

Hugh McKinstry is now serving as first vice-president of the Society of Economic Geologists. He is one of eleven AIME Members now directing the Society. The others are: **Olaf N. Rove**, Secretary; **J. T. Singewald, Jr.**, treasurer; and Councilors **Ernest F. Bean**; **Gilbert H. Cady**; **Carlton D. Hulin**; **Donald Davidson**; **William Callahan**; **Philip J. Shenon**; **Andrew Leith** and **John Gustafson**.

Arnold H. Miller, consulting engineer with offices in New York City, has been appointed by the board of directors as consulting engineer for the newly formed Cia. Minera Real de Plomosas, S.A. de C. V. Mr. Miller is at present in Sinaloa, Mexico, on work for the Company.

James J. Naughten is now employed as an assistant engineer with the engineering department of the Anaconda Copper Mining Co.

Earl K. Nixon took a three month leave of absence from the Kansas State Geological Survey during the summer to go to Alaska and the Yukon on private consulting work, evaluating mineral deposits of various kinds.

John C. Nixon received the degree of Doctor of Philosophy in metallurgy from the University of Utah last June for research on the theory of flotation. He is working at present for the Climax Molybdenum Co. in Climax, Colo., and hopes to return to his home in Australia before the end of the year.

Peter B. Nalle, after receiving his M.S. degree in mining from Columbia in January, started work for the St. Joseph Lead Co. at Bonne Terre.

Charles A. O'Connell is now underground manager, having been promoted from mine engineer, for Mufulira Copper Mines, Mufulira, Northern Rhodesia, S. Africa.

Edward B. Olds is addressed at the Bunker Hill & Sullivan Mining and Concentrating Co., Kellogg, Idaho. He had been with Consolidated Coppermines Corp.

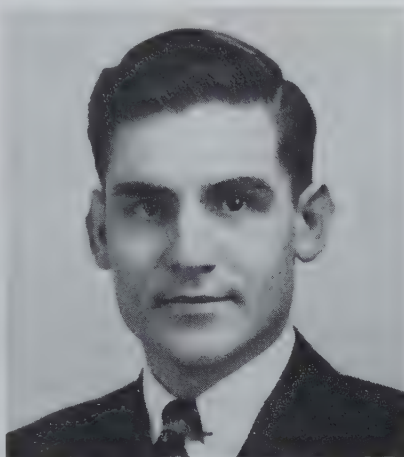
T. A. O'Hara has changed his address from Bissett, Man., where he was employed by San Antonio Gold Mines, to Flin Flon, Man. He is now employed as mining engineer by the Hudson Bay Mining and Smelting Co. with work as assistant to the testing and ventilation engineers. He has been testing and reporting on the performance of the low carbon nickel alloys drill steel which the Company hopes will replace the present high carbon alloy drill steel in current use.

A. A. Parish, formerly with Australian Iron and Steel Ltd., has become works manager for Southern Portland Cement Ltd., limestone

quarrying and cement manufacturing, Moss Vale, N.S.W., Australia.

D. W. Phillips has been appointed to the chair of mining at the New South Wales University of Technology, Broadway, Sydney, Australia.

Robert N. Pursel, after leaving military service in 1946, returned to Venezuela with the Texas Petroleum Co. where he was on the engineering staff. On June 16 he left for the Middle East for engineering work on the Trans Arabia Pipeline Company's pipe line from Saudi Arabia to the Mediterranean Sea. His new address is in care of the Company, P. O. 1348, Beirut, Lebanon.



Francis P. Williams, Jr.

Francis P. Williams, Jr., who graduated from Tri-State College last March with a degree in chemical engineering, is now employed as a chemist with the experimental division, Western Precipitation Corp., 1016 W. 9th St., Los Angeles, Calif. Before completing his college work, Mr. Williams had served for two years with the Navy in the Pacific, and prior to that, was with the Nevada-Massachusetts Co.

Thomas A. A. Quarm is no longer employed by the Frontino Gold Mines, Ltd., Antioquia, Colombia, where he was shift boss in charge of smelting. He is now with the Cerro de Pasco Copper Corp. as assistant research metallurgist. He receives mail at the Hotel Junin, La Oroya, Lima, Peru.

John G. Reilly has resigned his post as general manager of the Bayard department of the U. S. Smelting Refining and Mining Co. to become a consultant. He will continue to reside at 407 College Avenue, Silver City, N. Mex.

H. A. Quinn has returned from West Africa and resumed his former post on the geological staff of Norman W. Byrne, consulting mining engineer, at Yellowknife, N.W.T.

August F. Rambosek is now with the Phelps Dodge Corp. in Tyronne, New Mexico, having moved there from Park City, Utah. Mr. Rambosek, who was married a few months ago, is a graduate of the Montana School of Mines.

Francis F. Redfield has returned to the States from Aguilar, Argentina, and is now working as a mining engineer with the St. Joseph Lead Co.

John J. Reed in September of 1947 was transferred by the Braden Copper Co. from his post as assistant mine foreman and since that time has been field engineer in the hydraulic division of the electrical department. His present work involves the operation, maintenance, and construction of the extensive hydro electric power system developed by Braden. Recently, Mr. Reed returned to Chile after spending a four month vacation with his family.

John A. Riddle, after graduation from the Colorado School of Mines in May, took the job of an engineering trainee with the Oliver Iron Mining Co., Coleraine, Minn.

John A. Riley was named president of the Colombian Development Corp., New York City, at the concern's May board meeting.

Vernon J. Rogers, formerly at Washington State College, is now field assistant with the U. S. Geological Survey and receives his mail at General Delivery, Bozeman, Mont.

Robert L. Root completed the course in mining geology at the Missouri School of Mines and Metallurgy in January and is now working as assistant engineer and geologist for the International Smelting and Refining Co.

John W. F. Rudnicki received a B.S. degree in mining engineering in 1948 from Lehigh and a B.S. in civil engineering from the same university this February. His present post is field engineer in the Chesapeake and Ohio Railway coal development department in Huntington, W. Va.

Carl F. Schaber is now with the Mackenzie Engineering Co., 8 Dake St., Manchester Square, London W-1, as resident engineer in charge of building the new 500,000 ton steel plant in Yugoslavia. He had been a member of the staff of H. A. Brassert & Co. His permanent address remains at 500 S. Tin Ave., Deming, N. Mex.

Edward A. Sawitzke is now an engineer with the E. J. Longyear Co., Minneapolis, Minn., having left his post as secretary and vice-president of the Diamond Drilling Co. In his new job, he's working on the Fried-

ensville shaft in Friedensville, Pa. Letters reach him at Box 54, Center Valley, Pa.

A. H. Shoemaker, general manager of the Triumph Mining Co., was elected president of the Idaho Mining Ass'n at its annual meeting in June. At the same time **Henry L. Day**, president and general manager of Day Mines, Inc., was elected vice-president of the Association, and **Harry W. Marsh** of Boise was re-elected secretary. **Harry D. Bailey** of Stibnite, resident manager of the Bradley Mining Co., and **J. C. Kieffer** of Kellogg, manager of the Spokane-Idaho Mining Co., will serve as two of the four directors elected for the forthcoming year.

E. Nelson Strang, who graduated in June from Lafayette College, and who was president of the John Markle Society at that school, is now a mine inspector for the Stonega Coal and Coke Co. in Big Stone Gap, Va. Mr. Strang was a transitman for the mine from 1938 to 1942, and during his summer vacations in 1946, '47 and '48.

— At the Rio Conference —

When the First Pan American Engineering Congress met in Rio de Janeiro from July 15 to 24th, there were fourteen AIME Members there to present technical papers. Those who attended were:

T. G. MacKenzie, who collaborated on a paper on the economics of power development in Latin America; **Richard J. Lund**, who presented his paper on "Stockpiling in the U. S.—Past, Present and Future" (which appeared in the August issue of ME); **Sherwin F. Kelly**, who spoke on geophysical methods applied to exploration for ground water supplies; **J. H. Evans**, "Geophysical Explorations for Mineral Deposits"; **H. W. Straley, III**, "Copper Deposits of the Western Hemisphere"; **R. L. Anderson**, "Trends in Mechanical Loading and Strip Mining of Bituminous Coal"; **W. A. Leech, Jr.**, "By-Product Coking"; **Richard M. Foose**, "Photogeology—A Tool for Mineral Development"; **A. M. Bateman**, "America's Stake in the Mineral Resources of the World" (which appeared in the July issue of ME); **J. M. Wolfe** and **Fred T. Agthe**, "Modern Portland Cement Practice"; **Harry H. Power**, "Petroleum Engineering Education and the Quantitative Approach"; **John A. Ruppert, Jr.**, and **John E. Conley**, "Developments in the Manufacture of Lightweight Aggregates"; and **Carl W. Westphal**, "Modern Mechanical Methods in the Chemical and Processing Industries."



George M. Potter

George M. Potter has completed his one year assignment with the Foreign Minerals Branch of the Bureau of Mines in Mexico and has returned to the Salt Lake City branch of the metallurgical division. His new address is Bureau of Mines, 1600 E. First S. St., Salt Lake City 1.

Herbert C. Schweitzer is with the Molybdenite Corp. of Canada, La Corne, Val D'or, Que. He was previously employed by the Associated Metals and Minerals Corp.

Robert H. Shepard graduated in June from Brooklyn College with a B.S. degree in geology. He is employed at present in the mineral classification branch, conservation division, of the U. S. Geological Survey. He can be addressed care of the Survey, P. O. Box 997, Roswell, N. Mex.

Robert L. Swain became assistant chief mine engineer of the Braden Copper Co., Rancagua, Chile, and can be reached at the mine staff house, care of the Company. He had been with the Consolidated Coppermines Corp.

John S. Sumner, who recently graduated from the University of Minnesota with a B.S. degree in geology and physics, is now working as an assistant geologist with the Cleveland-Cliffs Iron Co., Ishpeming, Mich.

H. A. Toelle, Jr., can be reached in care of the Braden Copper Co., Rancagua, Chile. He had been addressed at Michigan College of Mining and Technology.

Paul M. Tyler is now in Europe studying ECA activities as part of his new position as minerals consultant for the Joint Congressional Committee on Foreign Economic Cooperation. He expects to visit several projects in the Near East and Africa, and then return to Washington in the fall.

William Huff Wagner, consulting engineer, is now secretary of the new Committee on Engineers in Civil Service of the Engineers Joint Council. The five-man Committee is composed of representatives from ASCE, ASChE, ASME, AIEE, and AIME, represented by Mr. Wagner. Its function is to maintain close liaison with the U. S. Civil Service Commission and other Federal agencies, in order to provide closer cooperation between the government and the 100,000 members of the four Founder Societies and the AICChE. Questions such as those of job standards and qualifications of government-employed engineers will form an important part of the Committee's work. Present headquarters are in the Washington offices of the ASCE.

Louis Ware, president of the International Minerals and Chemical Corp., sailed for Europe on July 12th for a two month business trip, with the object of studying economic conditions on the Continent and inspecting French and German potash mines in which his company has interests. Accompanied by Mrs. Ware, Mr. Ware visited European agents and customers of the Company.

Woodrow W. West has been appointed chief sales engineer for the Pennsylvania Crusher Company's division of bath iron works in Philadelphia. Mr. West has been with the company for seven years, working up through engineering, testing, shop inspection, and sales. He had a part in engineering and designing the Reversible impactor, a device which has begun to replace the standard type hammermill in the cement and stone industries. Mr. West has studied engineering at both Indiana Technical College and Temple University.

G. M. Wiles has been appointed division manager of the St. Louis smelting and refining division of the National Lead Co., and **A. R. Reiser** has been named as his assistant. At the same time, two other executive changes have made **H. A. Krueger** plant manager of the Tri-State operations of the division, and **A. J. Yahn** plant manager of the division's Frederickstown operation. Other changes in the Company now find **Frank R. Milliken** serving as assistant manager of National Lead's Titanium Division, with headquarters in New York; **George W. Wunder** succeeding Mr. Milliken as manager of the MacIntyre development in Tahawus, N. Y.; and **Paul W. Allen** becoming assistant plant manager at that site.

J. H. Williams is now a fuel engineer on the staff of the Fairmont Coal Bureau. During the early years

of his career he was closely connected with the bituminous coal industry as a test engineer in Cleveland, and during the war engineered and operated several Ordnance Division plants. He has been a consulting engineer in the power field since that time.

Sidney E. Worthen is now a sectional engineer for Patino Mines and Enterprises Cons., and can be reached at Llallagua, Bolivia, S.A.

—In the Metals Branch—

Robert K. Allen and **Alvin H. Kasberg**, both seniors in the department of metallurgy at the University of Wisconsin, took top honors in this year's James F. Lincoln Arc Welding Foundation undergraduate awards, with their paper entitled "A Study of the Welding Characteristics of Aluminum Bronze Electrodes." The award, made on July 9, was \$1,000, and, in addition, an equal amount was presented to the University to establish four annual scholarships of \$250, to be known as the Robert K. Allen, Alvin H. Kasberg-Lincoln Foundation Scholarship. The award formed part of scholarships totalling \$6750 presented to three schools, and to 77 undergraduates in 47 engineering schools, which prizes are designed to stimulate engineering undergraduates to aid in the development of the science of arc welding.

George Allison is working at Dumas, Texas, as plant metallurgist for the American Zinc Co. of Illinois.

J. K. L. Andersen since June 1 has been employed as assistant to the director of A/S Bremanger Kraftsel-skap, a pig iron plant, boks 524, Bergen, Norway.

Edward L. AuBuchon, since his graduation from the Missouri School of Mines last May, has taken the job of open-hearth metallurgist with the Wisconsin Steel Works, Chicago. His address is 10232 S. Ave. J, Chicago 17.

Edgar C. Bain, vice-president of the Carnegie-Illinois Steel Co., will receive the 1949 Gold Medal of the American Society for Metals at the annual dinner of the Society on Oct. 20 at the Hotel Statler, Cleveland. Dr. Bain receives this, one of the highest honors made available in the metals industry, in recognition of his great versatility in applying science to the metal industry.

George M. Baumann has been made purchasing agent and assistant to the plant manager of the Whiting plant, Federated Metals Division, American Smelting and Refining Co. Associated with Federated Metals since 1936, he has served in various

capacities in various plants in the States and in Mexico. For the past three years he had been assistant to the general manager of scrap purchases at the Company's headquarters in New York City.

J. H. Bechtold is research metallurgist for the Westinghouse Research Laboratories at East Pittsburgh, Pa. His address is 5801 Aylesboro Ave., Pittsburgh 17.

George A. Bivens, having graduated from the University of Michigan, is a junior engineer with the Worthington Pump and Machinery Corp., Harrison, N. J.

John B. Campbell is associate editor of **Materials and Methods**, Reinhold Publishing Corp., 330 W. 42nd St., New York City 18.

Kenneth L. M. Dodd is now managing the Vancouver office of Canadian Allis-Chalmers, Ltd. He was formerly a field engineer with the Denver Equipment Co.



Harley S. Van Fleet

Harley S. Van Fleet has been appointed manager of Atlantic division research for the American Can Co., with headquarters in New York City. He was formerly chief of the container research section at the Company's general research laboratory.

John F. Elliott is now with the research laboratories of the U. S. Steel Corp. in Kearny, N. J. Mr. Elliott took his Sc.D. degree in metallurgy from MIT in June, having written his doctor's thesis on "The Thermodynamic Properties of Liquid Metallic Solutions."

George Enzian became assistant manager of metallurgical research of the Jones & Laughlin Steel Corp., Pittsburgh, on May 1. He is secretary of the Pittsburgh Section, AIME, as well as being active in the Pittsburgh Local Section of the Open Hearth Committee.



Carl Zapffe

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Carl A. Zapffe and **Miss M. E. Haslem** have been awarded a Certificate of Honorable Mention by the Wire Association for presenting the outstanding paper in the ferrous division of that industry in 1948. The paper described the discovery that hydrogen gas is absorbed by certain steels from inhibited pickling solutions in quantities as great as from uninhibited acids, causing important damage which has previously been blamed on other factors. Dr. Zapffe and Miss Haslem will receive the award at the annual banquet of the Wire Assn. in Chicago on Oct. 19.

Arthur E. Focke, research metallurgist for the Diamond Chain Co., Indianapolis, has been nominated to serve for one year beginning next fall as president of the American Society for Metals. **Elmer Gammeter**, chief metallurgist for Globe Steel Tubes Co. of Milwaukee, has been nominated to the board of trustees for a two-year term. Dr. Focke is a graduate of Ohio State University, where he also received his M.S. and Ph.D. degrees. Prior to his Diamond Chain affiliation, which began in 1930, he had been special metallurgist for General Electric Co. and chief engineer for P. R. Mallory Co. Mr. Gammeter, an employee of Globe Steel Tubes since 1943, had previously been manager of the stainless steel bureau, metallurgical division, Carnegie-Illinois Steel Corp. in Chicago, which followed six years as metallurgist for Edison General Electric Appliance Co.

O. B. J. Fraser, of the International Nickel Co. in New York, has been named president of the American Welding Society for the coming year.

Norman C. Jensen is an engineer with Consolidated Builders, Inc., at

the Detroit Dam. His new address is Box 515, Mill City, Oreg.

David W. Levinson has been appointed an instructor in the department of metallurgical engineering at Illinois Institute of Technology in Chicago. Mr. Levinson, who assumed his new duties on Sept. 1, took his master's degree at Tech last June, and received his bachelor's degree in February of last year. He will work under **Otto Zmeskal**, director of the department at the school.

S. C. Massari has been awarded the John H. Whiting Gold Medal of the American Foundrymen's Society, for "outstanding contributions in the field of ferrous metallurgy, molding and foundry practice and service to the wartime foundry industry with the Chicago Ordnance District." Mr. Massari is technical director of the AFS and was for 18 years research metallurgist for the Association of Chilled Car Wheel Mfrs.

Donald J. McMaster is a trainee in the remelt division of the Permanente Metals Corp. at Trentwood, Wash. His address there is N4501 Bannon Ct.

Ralph W. Preston, Jr., is now employed by the Timken Roller Bearing Co. as a metallurgist trainee. He can be reached at the YMCA, 405 2nd St., N.W., Canton 2, Ohio.

Raymond A. Quadt has been appointed director of aluminum development for the Federated Metals Division of the American Smelting and Refining Co. in Detroit. Mr. Quadt, who was formerly assistant manager of the firm's general aluminum department, will continue his extensive aluminum research in his new position—which research has already led to the development of Tenzaloy.

Gilbert Soler, of the Iron and Steel Division, AIME, is now vice-president of manufacturing operations for Atlas Steels, Ltd., Welland, Ont., Canada. He was formerly works manager for the Company.

J. C. Warner, dean of graduate studies and head of the chemistry department at Carnegie Institute of Technology, has been named president-elect of Carnegie Institute. He will fill a newly created office of vice-president until his inauguration after the present president retires on July 1, 1950. Dr. Warner's election climaxes 23 years of outstanding service on the Carnegie faculty. Starting out as an instructor in chemistry in 1926, he advanced up the academic ranks to become professor of chemistry and head of that department in 1938 and dean of graduate studies in 1945. On a two-year war

leave from the campus, as one of the nation-wide team of scientists which turned out the atomic bomb, he directed and co-ordinated research in the laboratories at the Universities of California and Chicago, Iowa State, MIT, and at Los Alamos, in highly confidential work on the purification of plutonium.

—In Petroleum Circles—

R. C. "Bob" Chenoweth has become manager of machinery sales for the Republic Supply Co. with headquarters in Houston. He has been engaged in the sales of machinery to



R. C. Chenoweth

the oil industry since 1929, at which time he started selling pumps in the Mid-Continent territory following graduation in mechanical engineering from Purdue. Since 1939 he has been located in Houston and has represented several of the large machinery accounts in the Gulf Coast and Mexico areas.

Rodney R. Adams has finished his schooling at the University of New Hampshire and is working for the Seismograph Service Corp. of Tulsa.

Ralph F. Atkinson is a junior engineer in the production department of the British American Oil Co. in Edmonton, Alta.

James R. Cameron has joined the research staff of Battelle Memorial Institute, Columbus, Ohio, where he is doing research in nonferrous metallurgy. An Ohio State graduate, he was associated with the Stanolind Oil and Gas Co. in Gorham, Kans., before taking this job.

John M. Cooper is assistant managing director of the Kuwait Oil Co. with offices at 1 Great Cumberland Place, London W1, England. He had been with the Gulf Oil Corp. in Houston.

W. F. Dalton, who was chief engineer of the Hunt Oil Co., is now

president of the Placid Oil Co., Shreveport, La.

Donald M. Davidson has been elected vice-president of the E. J. Longyear Co., Minneapolis, diamond drill contractors and manufacturers.

Thornton Davis, president of the Linda Petroleum Co., San Antonio 5, Texas, was formerly vice-president and manager of the Peerless Oil and Gas Co.

Everette L. De Golyer received an honorary degree of Doctor of Engineering at Princeton University's 202nd annual commencement on June 14. The citation characterized him as "a geologist and oil producer whose international reputation in the field of petroleum engineering is unexcelled," as well as "a prospector in the discovery of human abilities."

Elmer L. DeMaris is supervisor of exploration for the General Petroleum Corp., Box 2122, Terminal Annex, Los Angeles 54, Calif.

E. G. Dobson has been transferred to the post of chief petroleum engineer of the Texas Petroleum Co., Caracas, Venezuela. He had been in Bogota, Colombia.

William F. Ellis is assistant petroleum engineer with the Union Producing Co., P. O. Box 872, Vivian, La.

Glen E. Emmett, having graduated from Texas Technological College, has taken the job of engineer revenue agent with the Bureau of Internal Revenue. His new address is 2159 Lovedale Ave., Dallas 9, Texas.

L. W. Fagg is a partner in the Johnson-Fagg Engineering Co., 4020 S. Peoria Ave., Tulsa 5, Okla.

George H. Fentress is now a technical trainee in the geophysics section of the Phillips Petroleum Co. He received his degree in geological engineering from the Colorado School of Mines last May.

Ray L. Freeborn, who is with the Continental Oil Co., has been moved from Wichita Falls, Texas, to serve as district engineer in the organization's New Mexican District. He is addressed care of the Company, Box CC, Hobbs, N. Mex.

John C. Gullivan is now production superintendent for the Wood River Oil and Refining Co., with offices at 2527 20th St., Great Bend, Kans.

James H. Galloway is now district superintendent for the Humble Oil and Refining Co., at 612 S. Flower St., Los Angeles, Calif.

E. A. Galvin has joined the Western Gulf Oil Co., 1260 Terminal Bldg., Los Angeles, Calif., as chief production engineer.

Alvin A. Geyer, who had been studying at Louisiana State, is now a roustabout engineer with the Pan American Gas Co. at Texas City, Texas.

David Goodwill is now a production analyst in the production department of the Standard Oil Co. of California, at Box 151, Whittier, Calif.

Ralph C. Graham, who was chief geologist with the Tennessee Gas and Transmission Co., is vice-president of the Coast Co., P. O. Box 1181, Houston.

George H. Gray of Midland, Texas, has become division engineer for the Sinclair Prairie Oil Co., at P. O. Box 1470 in that city.

Russell W. Haller, after graduation from the University of Pittsburgh, joined the Continental Oil Co. as an engineer trainee. He is now working at the Company's Gebo, Wyo., field. Mr. Haller is addressed care of the Company, Thermopolis, Wyo.

Harold H. Knapp is a trainee engineer with the Ohio Oil Co. at Eureka, Kans. He had been a student at the University of Tulsa.

Dan Kralis has become manager of exploration and assistant to the president of the Sunland Refining Corp., in Fresno, Calif. He heads the newly-formed division of exploration for the Company, and is carrying out development and exploration work in Texas, the Rockies, California and Nevada. Mr. Kralis was formerly with the Petroleum Exploration Service, a consulting firm, as chief geologist. His new home address is 1307 Dakota Ave., Fresno.

Jean Warren Meyers, formerly with the Stanolind Oil and Gas Co., is working for the Champlin Refining Co., Ellinwood, Kans., as a petroleum engineer.

Paul O. Naut, no longer a student at Oklahoma University, is an engineering trainee with the Creole Petroleum Corp. at Tia Juana, Zulia, Venezuela.

John A. Poulin can be addressed Oil and Gas Division, Foreign Branch, U. S. Dept. of the Interior, Washington 25, D. C.

Joe Lee Terry is now with the geological department of the Houston Oil Co. of Texas, in Houston.

Lyon F. Terry has been made vice-president of the petroleum department of the Chase National Bank.

E. V. Watts has been appointed production superintendent of the Southern Division of the General Petroleum Corp.'s production department. He had been assistant to the southern division superintendent since May of last year. Mr. Watts has been with the Company since his graduation from the California Institute of Technology in 1936, when he began as a roustabout in the Lost Hills field. His home address is 529 Garfield Ave., S. Pasadena, Calif.

Obituaries

ROBERT H. JEFFREY (Member 1896) British-born mining engineer who had spent most of his career in Mexico, died on Jan. 31, at the age of 75. Mr. Jeffrey was a graduate of the Royal School of Mines in England, and began his professional career in this country at mines in Virginia and Arizona. In



Robert H. Jeffrey

1898 he joined the Pinos Altos Co., in Chihuahua, Mexico, later working for the Avino Mines, Ltd., in Durango, and then beginning an unbroken association with the Mazapil Copper Co. in Saltillo, Coahuila. He retired in 1927, but was soon recalled, and made a director of the Company the following year. He also served as director of four railroads in Mexico. For many years Mr. Jeffrey served as British Vice-Consul in Saltillo, and, in 1914, when the U. S. troops entered Vera Cruz, he was in charge of the U. S. consulate there. Mr. Jeffrey claimed to have installed the first flotation table in Mexico, and had patents on both the Jeffrey automatic feeder and the Jeffrey concentrator.

James MacNaughton

An Appreciation by C. Harry Benedict

James MacNaughton, former president of the Calumet & Hecla Consolidated Copper Co., died at his home in Calumet on May 26. He was eighty-five years old and had been seriously ill for some years.

Mr. MacNaughton has taken an active part in Institute affairs throughout his professional career. Elected to membership in 1890, he was one of the oldest members of the Legion of Honor. From 1916 to 1918 he served as vice-president and in 1935 was the recipient of the William Lawrence Saunders medal.

Born March 9, 1864, at Bruce Mines, Ont., Canada, Mr. MacNaughton was brought to the Michigan copper country

at the early age of three months. His father took employment with the newly opened and rapidly developing Calumet & Hecla Mining companies, distinct corporations at that time, constructing milling facilities at Lake Linden. Young James himself at the age of eleven worked as water boy for the same company during his vacation, and again five years later at the completion of his primary education, as a stationary engineer.

His college career consisted of one year residence at Oberlin, followed by an engineering course at the University of Michigan from which he received the degree of B.S. in Civil Engineering with the class of 1888. In 1930 he was awarded the honorary degree of Doctor of Science by the Michigan College of Mining & Technology, on whose Board of Control he served for many years. But most of his education came from detailed experience in the field.

His rise in the mining world was a rapid one. After leaving college he returned first to his beloved Calumet & Hecla for a short time as junior engineer and in 1889 accepted a corresponding position with the Chapin mine at Iron Mountain, Mich. Within three years he was general manager of the Chapin and under his direction it became the largest producer of iron ore in the state, and equally famous for its efficiency and low cost of operation. He remained at the Chapin for twelve years.

At the turn of the century the Calumet & Hecla Copper Co. was facing a deepening and declining yield ore-body and Alexander Agassiz, president of that company, was looking about for a new manager to take charge. Mr. MacNaughton was a natural choice but it was a difficult decision for him to make in accepting the offer, for the iron ore industry was then entering a tremendous expansion. But Calumet was his home and the Calumet & Hecla his first love, and so in 1901 he came back as superintendent to the company from which he had earned his first dollar as water boy twenty-five years previously—truly an American saga.

Mr. MacNaughton had full responsibility for all operations at Calumet, many functions before his time having been taken care of at Boston. For forty years, under successive titles of general manager, vice-president and president, he guided the destinies of the company through an expansion period from the operation of a single mine to an integrated company with mining territory extending throughout three counties and employing more than 12,500 men. Under his leadership and because of the confidence that other mining and financial leaders reposed in him, the Calumet & Hecla for many years had a standing in industry out of proportion to its copper production.

In politics he was a staunch Republican and as such was a number of times delegate to Republican national conven-

tions and in 1900 a presidential elector. As befalls to all busy men Mr. MacNaughton devoted much of his time to civic and charitable activities in behalf of his employees and the community of which they and their families were the major part. His private beneficences were unostentatious, in keeping with his character.

The secret of Mr. MacNaughton's success as an executive and a leader of men lay in an engaging personality, a keen sense of humor, an uncanny ability to make rapid and correct decisions, and above all personal integrity. He had an inexhaustible fund of stories, mild for the ladies and pungent for the locker room. A pert phrase or the very pointed moral of a short story was used more often than heated argument to settle a controversy or gloss over a departmental dispute. Whether as considerate superior, genial host, or entertaining guest, his kindly nature will be long remembered and cherished by his neighbors and friends in the Copper Country, and his associates throughout the nation.

ROBERT HARDY BEDFORD (Member 1948), died in Los Gatos, Calif., on April 26, at the age of 66. Mr. Bedford, a consulting engineer, was born in New Zealand, and had a degree in general science from the University of New Zealand. He also attended the University of Missouri, receiving his B.Sc. degree from there in 1906, and an E.M. five years later. After several years in copper mining in Bisbee and Globe, Ariz., he became superintendent of the North Star mines in Grass Valley, Calif., remaining there for fifteen years. Mr. Bedford then set up a consulting practice, and was a consultant on gold mining for the Russian government during 1930-32. At the time of his death, he was a consultant for the Yellow Jacket Mining Co.

CHARLES A. BRUCE (Member 1948), a petroleum engineer, died on March 11 at Weeks Island, La., of accidental drowning. He was 28 years old. Mr. Bruce entered the Missouri School of Mines and Metallurgy in 1939, but his education was interrupted two years later, when he went to serve for four years in the Navy Air Corps. He took his B.S. degree in 1947, and was employed, at the time of his death, by the Shell Oil Co.

WILLIAM H. CORBOULD (Member 1892), a leading figure in Australian mining, and a Life Member of the AIME, died on March 16, at the age of 82. Mr. Corbould, a mining engineer and metallurgical chemist, was educated at Ballarat College, worked on the Broken Hill silver fields in 1885, and later traveled through England, Austria, Japan, France, Canada, Germany, and China as well as the U. S. A., studying mining geological formations, and metallurgical works.

In 1893 he walked from Freemantle, in

Western Australia, to the Coolgardie goldfields, a distance of 300 miles. It was this sort of determination which made him the directing spirit of Mt. Isa Mines, Ltd., in its early period. Mr. Corbould had the technical knowledge plus sufficient imagination to see the wealth of Mt. Isa's ore, and went on to implement his realization that only millions of dollars could successfully exploit the low-grade deposits. The organization which he so successfully brought to the world's attention is now worth more than \$17,000,000, exclusive of the ore bodies, the extent of which has not yet been delimited.



William J. Flori

WILLIAM J. FLORI (Member 1948), a junior mining engineer for the Miami Copper Co., died on May 7, at the age of 25. Mr. Flori attended Blackburn College from 1941 to 1943, and in that year became an aviation cadet in the U. S. Navy Air Corps. He rose to the rank of Ensign, was discharged in 1946, and completed his professional education at the Missouri School of Mines in June of last year. It was then that he joined Miami Copper.

R. CLYDE BUTLER (Member 1947), vice-president in charge of operations of the Empire Steel Co., died on March 28 at the age of 56. Mr. Butler was a native of Ohio, and graduated from Youngstown College in 1916. For four years he was a chemist for the Ohio Iron and Steel Co., then joined the Sharon Steel Corp., where he served for fourteen years as assistant superintendent of blast furnaces. In 1936 he joined the Pittsburgh Steel Co. as superintendent of blast furnaces, and moved up, within ten years, to the position of general manager for the firm. He was with the Steel Co. of Canada for a time before joining Empire Steel.

Necrology

Date Elected	Name	Date of Death
1920	John R. Comstock	July 14, 1949
1913	Emil Gathmann, Sr.	Aug. 23, 1949
1918	Joseph S. Henry	March 26, 1949
1937	Louis W. Huber	Aug. 17, 1949
1928	William D. Mark	June 16, 1949

JOSEPH P. LABAW (Member 1920), consulting mining engineer of Hopewell, N. J., died on April 27. He was 69 years old. Mr. Labaw received his degree in mining engineering from the Michigan College of Mines in 1903, and went on to work with many mining companies in all parts of this nation before he began his own consulting practice. His consulting work took him through Mexico, Canada, South America, and Costa Rica. His last position, before a heart condition forced him from more active work, was with the U. S. Metals Reserve, in the bauxite area of Arkansas, during the recent war.

JAMES E. LITTLE (Member 1909), formerly with the mining department of the Bethlehem Steel Co., died on May 24 after a brief illness. He was a graduate of Lehigh University, class of 1894, with a degree in mechanical engineering. His first eight years were spent with the Bethlehem Iron Co., predecessor of Bethlehem Steel. In 1902 he went with the Pennsylvania Steel Co., serving for ten years as a mechanical engineer with their Cuban subsidiary, the Spanish American Iron Co. As such, he engineered the mine development and shipping facilities at the Mayari iron ore deposits in Felton, Cuba. When the Company was acquired by Bethlehem Steel in 1916, he moved to Bethlehem, serving in the mining department until 1940, and in the research department until 1947, at which time he retired.

CARL H. LOUX (Member 1920), mining engineer and former superintendent for the Alan Wood Steel Co., Conshohocken, Pa., died on March 31, at the age of 60. Born in Idaho, Mr. Loux had studied at the University of California, and graduated in 1912 from the University of Idaho. Mr. Loux's early years were spent at various properties in Idaho, Montana, and up in Canada. At one time he had been assistant city engineer for the city of Pocatello, Idaho. Later he worked as a chemist for Anaconda, as an oil geologist for the Northern Pacific Railroad, and as superintendent for the Warren Foundry and Pipe Corp. in Oxford, N. J. He had also done special investigation of ore classifications at Anaconda, and designed a new mining method for the Washington mine in Oxford, N. J.

LEONARD LYNCH (Member 1947), a metallographist with the Wisconsin Steel Works of the International Harvester Co., died last Oct. 18 at the age of 35. Mr. Lynch attended Hibbing and Itasca Junior Colleges, and took a bachelor of chemistry degree from the University of Minnesota in 1936. The following year, he joined the McCormick works of International Harvester, and became control metallurgist and metallographist at the steel works in 1941.

William Clifton Phalen

An Appreciation by Oliver Bowles

W. C. Phalen, a member of the Institute since 1912, passed on at his home in Washington, D. C., on May 27, 1949. Born at Gloucester, Mass., in 1877, he received his B.S. degree in chemistry and M. S. in geology at MIT. He later was awarded a Ph.D. degree at George Washington University. He began his scientific career at the National Museum in 1902, and joined the staff of the U. S. Geological Survey in 1904. He served as a mineral technologist for the Bureau of Mines from 1916 to 1920. Part of this period was devoted to problems of strategic minerals essential to the conduct of the first World War.

Dr. Phalen's specialty was saline minerals, and because of his comprehensive knowledge of them he left the Bureau of Mines to become the salt specialist of the Solvay Process Co. at Syracuse, N. Y., which he served efficiently until his retirement in 1946. He was a part-time consultant for the Bureau of Mines at the time of his death.

Although quiet and unobtrusive, Dr. Phalen possessed a wealth of information on salines as well as on geology, mining, and chemistry in general. His wide knowledge, integrity, and quiet humor, attractive personality and loyal friendship endeared him to all of his associates.

E. PAUL KEUPER (Student 1948), a junior at the University of Cincinnati, died last December. Mr. Keuper, who was 21, expected to take his B.S. in metallurgical engineering in 1950.

CHARLES MENTZEL (Member 1909), a consulting mining engineer, died in New York City on June 1, at the age of sixty-seven. Since his graduation in 1907 from the Columbia School of Mines, Mr. Mentzel had been with mining enterprises in Canada, the Western United States, Mexico, and Panama. In 1914, he was a mine examiner for the Canadian government, and later, served in the same capacity in Mexico. In 1915, he opened manganese deposits in Mandinga Bay on the Gulf of San Blas, Panama. Mr. Mentzel was generally regarded as an expert on emeralds.

ARTHUR BERKELEY YATES (Member 1938), former chief geologist of the International Nickel Co., died in Montreal on May 10 at the age of 47. He had come to that city from South Africa to receive the Barlow Memorial Medal of the Canadian Institute of Mining and Metallurgy at their recent annual meeting.

Born in Lead, S. Dak., Dr. Yates took his B.Sc. from the University of California in 1922, and the M.Sc. and D.Sc. degrees from Harvard nine years later. From 1923-28 he was a surveyor, engineer, and geologist for the Homestake

Mining Co. Following two years as a research associate at Harvard, Dr. Yates joined International Nickel.



Arthur Berkeley Yates

He was a member of the CIM, a fellow of the Royal Society of Canada, and a member of the Society of Economic Geologists, and the Geological Society of America.

Harry H. Stout

AN APPRECIATION BY DONALD M. LIDDELL

Harry H. Stout died in Plainfield, N. J., on April 13, aged 76 years and four months, after a long, complicated illness, chiefly a severe arthritic condition. He was born in Sacaton, Ariz., Dec. 8, 1872. In 1878, his parents moved to Tidioute, Pa., and he entered the United States Military Academy in 1891 from Pennsylvania, graduating in 1895 and serving as Lieutenant in the Sixth Cavalry in the Spanish-American War. He resigned from the Army in 1901, and entered the chemical business in California, residing in San Francisco and Berkeley and serving as superintendent for the Peyton and the General Chemical Companies.

He was an ordnance officer with the American Expeditionary Force in France in World War I and was commended officially by General John J. Pershing. He became chief metallurgist for Phelps Dodge in 1921 and retired in 1931. The rejuvenation of the Nichols Copper works was one of his achievements. Before his retirement, he had lived at Ardsley-on-the-Hudson but moved shortly afterward to Plainfield.

He had been a member of the AIME for about 32 years. The Secretary remembers a session at the Engineers' Club with the late Colonel Barbour and Colonel Stout on the eve of his sailing for France in World War I and Colonel Stout's writing down an equation: the value of an engineer = professional knowledge $\times$ honesty² $\times$ guts³ $\times$ loyalty⁴.

He had these qualities without the necessity of any exponents.

HUBERT MERRYWEATHER (Member 1916), retired Bethlehem Steel Co. executive, died of a heart attack on June 7, at the age of 67. When he retired in 1947, Mr. Merryweather was general manager of ore properties for the Company, having been with Bethlehem Steel for 36 years. After graduating from MIT in 1904, he spent six years in mining activities in the West and at various sites in Mexico. Then he took over as mine superintendent for Bethlehem's Juragua Iron Mines Co., in Cuba. After three years he was sent to Chile, to become vice-president and general manager for the Tofo iron mines there. Later, he returned to Cuba as vice-president and general manager of the Bethlehem-Cuba Mines Co., remaining there until 1928, when he was recalled to Bethlehem. He was made general manager of ore properties in 1939.

RUSSELL E. REILLY (Member 1945), manager of the Wyodak Coal and Mfg. Co., Gillette, Wyo., is dead. Mr. Reilly had been with the Company since 1941, and prior to that had spent twelve years in various positions with the Homestake Mining Co., ending there as head of the water and property department. A graduate of South Dakota's State Normal School, Mr. Reilly spent his first fifteen years with Homestake, and, from 1923 to 1929 was in charge of Cerro de Pasco's engineering office in Peru.

CHARD O. SANFORD (Member 1917), consulting mining engineer of Washington, D. C., died on Jan. 31 at the age of 66. In addition to his consulting practice, Mr. Sanford had been a mining engineer with the Bureau of Mines, the RFC, and the Premium Price Plan for metals from 1942 to 1947. Born in Iowa in 1884, Mr. Sanford attended the Universities of Washington, California, and Arizona, beginning his career as an assayer with the Skidoo Mines Co. in Inyo, Calif. In 1914 he was superintendent of the Antequera Tin Mine in Paza, Bolivia, and, fourteen years later, held the same position with the Tom Reed mine in Oatman, Ariz. During World War I Mr. Sanford was a First Lieutenant with the Corps of Engineers. In 1938 he was Secretary of the Los Angeles Section, AIME.

GEORGE L. SMITH (Member 1945) is dead. Mr. Smith had spent his whole professional career with the Rochester and Pittsburg Coal Co., Indiana, Pa. He joined the company as a laborer a year after graduation from the Clarkson College of Technology in 1917, and through the years rose to the position of operating vice-president, which job he held at the time of his death.

PHILIP S. SMITH (Member 1918), leading government geologist, and a prolific writer in his field, died on May 10 at the age of 71. Mr. Smith had retired in 1946, but had since then been actively

engaged in private exploration work, chiefly in Alaska. He had three degrees, A.B., A.M., and Ph.D., from Harvard University. After receiving his A.B. in 1899, he joined the U. S. Geological Survey as a geologic aid. Two years later he was an instructor in geology and mining at Harvard, this association lasting for four years. Returning to the USGS, he began work on Alaskan explorations, and by 1925 he was named chief Alaskan geologist, continuing as such until his retirement three years ago. During that time he made numerous special investigations in the areal, physiographic, and economic geology of Alaska. One of the results of his work was the total of over 100 reports, published by the USGS, and in technical journals and publications of scientific societies.

Mr. Smith was a member of over seventeen professional organizations, including the Washington Academy of Sciences, the Society of Economic Geolo-

gists, the American Geophysical Union, and the American Polar Society.

WALTER STALDER (Member 1915), consulting petroleum geologist in San Francisco, died on May 27 at the age of 67. Mr. Stalder, who had been in consulting work since 1915, held B.S. and M.S. degrees from the University of California, the latter having been granted in 1907. Before entering private practice he had been in charge of property evaluation for the Union Oil Co. of California, and worked with M. L. Requa; the San Francisco Exploration Co.; and the Nevada Petroleum Co. Early in his career he developed the Decrease Curve method of estimating oil reserves, and later, in 1933, brought in the Sutter Buttes gas field in California. Mr. Stalder was the author of numerous articles on California oil geology which appeared in California state bulletins, professional magazines, and newspapers.

Proposed for Membership

Total AIME membership on July 31, 1949, was 15,844; in addition 4431 Student Associates were enrolled.

ADMISSIONS COMMITTEE

James L. Head, Chairman; Albert J. Phillips, Vice-Chairman; George B. Corless, T. B. Counselman, Ivan A. Given, George C. Heikes, Richard D. Mollison, and Philip D. Wilson.

Institute members are urged to review this list as soon as the issue is received and immediately

to wire the Secretary's office, night message collect, if objection is offered to the admission of any applicant. Details of the objection should follow by air mail. The Institute desires to extend its privileges to every person to whom it can be of service but does not desire to admit persons unless they are qualified.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; AM, Associate Member; S, Student Associate; F, Junior Foreign Affiliate.

ALABAMA

Sheffield—TIEMANN, THEODORE DONALD. (M). Research engineer, Reynolds Metals Co.

University—B A T O R, GEORGE THOMAS. (C/S—J-M). Associate professor of mining, University of Alabama.

ARIZONA

Ajo—RUSK, ALBERT T. (C/S—J-M). Mining engineer, New Cornelia branch, Phelps Dodge Corp.

Globe—MESSER, BEN GRANT. (C/S—J-M). Chief mine engineer, Miami Copper Co.

Inspiration—HAMBURGER, RICHARD. (C/S—J-M). Junior engineer, Inspiration Consolidated Copper Co.

CALIFORNIA

Arcadia—MAYHEW, ELDON JAY. (C/S—J-M). Operations manager, Building Products Div., Great Lakes Carbon Co.

Bakersfield—DORWART, GEORGE MARTIN. (C/S—J-M). Production foreman, Union Oil Co. of California.

Betteravia—GALLOWAY, TORRENCE DELOSS. (C/S—J-M). Research engineer, Union Sugar Co.

Darwin—TOGNONI, HALE, CHRISTOPHER. (C/S—S-J). Stope geologist and sampler, Anaconda Copper Mining Co.

Long Beach—ENSLEY, CHARLES, EARL. (C/S—J-M). Development en-

gineer, Southern Division, Richfield Oil Corp.; **STONE**, REID TOLAN. (C/S—S-J). Assistant field engineer, Axelson Manufacturing Co.; **THORLEY**, THOMAS J. (C/S—J-M). Senior harbor engineer, Port of Long Beach, Calif.

Los Angeles—BUTLER, WALLACE PIERCE. (C/S—J-M). President, Butler Ore Co.; **MEYER**, GENE. (C/S—J-M). Mill superintendent, Cie. Aramayo de Mines en Bolivie.

Oakland—OAKESHOTT, GORDON BLAISDELL. (M). Associate geologist, California Division of Mines.

Palo Alto—HURST, GEORGE PATTERSON. (AM). Sales engineer, Bethlehem Steel Co., San Francisco Yard.

Riverside—MILLS, EUGENE ARTHUR. (C/S—J-M). Mining engineer in charge, Oliver Iron Mining Co. of Venezuela.

Sacramento—CRAIG, ROBERT HITCHCOCK. (C/S—J-M). Civil engineer, Corps of Engineers.

San Francisco—PRIMM, HERBERT ENGLAND. (M). Sales representative, Harbison-Walker Refractories Co.; **RAWLINGS**, STUART LAMAR, JR. (M). Vice-president and director, Standard Cyaniding Co. Director, San Luis Mining Co.

San Mateo—COULTER, RONALD SCOTT. (M). Combustion engineer, Pacific Coast operations, Bethlehem Pacific Coast Steel Corp.

Seal Beach—WALKER, ROBERT CARLTON. (C/S—S-J). Junior engineer, General Petroleum Co.

Whittier—ARTHUR, MILAN G. (C/S—J-M). Division production engineer, Union Oil Company of California.

COLORADO

Denver—NORMAN, TELFER E. (C/S—J-M). Metallurgical engineer, Climax Molybdenum Co.; **RIDDLE**, JOHN ALDEN. (C/S—S-J). Engineer trainee, Oliver Iron Mining Co.; **WILSON**, MALCOLM E. (C/S—J-M). Assistant to manager, wire products sales, Colorado Fuel & Iron Corp.

Gilman—SKINNER, JOHN PERKINS. (C/S—J-M). Assistant mine foreman, New Jersey Zinc Co.

Golden—CARPENTER, ROBERT H. (C/S—J-M). Assistant professor, Colorado School of Mines; consultant, New York & Honduras Rosario Mining Co.; **ROBERTS**, FRANK CULMER. (C/S—J-M). Design engineer in charge of Golden office, Climax Molybdenum Co.

Grand Junction—HALL, ROBERT B. (C/S—J-M). Geologist, Mineral deposits branch, U. S. Geological Survey.

Leadville—GUNELSON, ADOLPH GERHARD. (R, C/S—J-M). Chief mine engineer, Resurrection Mining Co.

Ouray—UNGER, RICHARD WILSON. (C/S—J-M). Mill superintendent, Idarado Mining Co.

Rifle—WRIGHT, FREDERICK DUNSTAN. (C/S—J-M). Unit chief of mining investigations unit, U. S. Bureau of Mines.

CONNECTICUT

New Haven—KOLB, THOMAS THORNTON. (C/S—J-M). General superintendent, A. N. Farnham, Inc.

DISTRICT OF COLUMBIA

Washington—FICK, NATHANIEL CROW. (C/S—J-M). Metallurgist and deputy executive director of Committee on Basic Physical Sciences, Dept. of National Defense.

IDAHO

Burke—FOLWELL, WILLIAM THOMAS. (C/S—J-M). Mining engineer, Hecla Mining Co.

Patterson—MECIA, JOSEPH ANTHONY. (C/S—J-M). Manager, Ima Mine, Bradley Mining Co.

ILLINOIS

Chicago—MANNAS, WILLIAM JOSEPH. (C/S—S-J). Petroleum engineer, The Pure Oil Co.; **NICKEL**, MELVIN E. (C/S—J-M). Assistant superintendent, open hearths, Wisconsin Steel Works, International Harvester Co.; **WOODS**, HENRY COCHRANE. (M). Chairman of board and vice-president, Sahara Coal Co.

Peoria—ALBERS, FRANCIS CLINTON. (C/S—J-M). Staff metallurgist, Caterpillar Tractor Co.; **BESS-**

LER, DELMAR RUSSELL. (M). Operating metallurgist, Keystone Steel & Wire Co.; **REYER, EDWARD HARRY.** (M). Open hearth superintendent, Keystone Steel & Wire Co.

INDIANA

East Chicago—**DOAN, DONALD JAY.** (C/S—J-M). Research metallurgist, Eagle-Picher Co.; **RASSENFOSS, JOHN ALBERT.** (C/S—J-M). Research metallurgist, American Steel Foundries.

West Lafayette—**TAYLOR, WILLIAM EDWIN.** (C/S—S-J). Research fellow, Purdue University.

KANSAS

Hugoton—**FLOOD, WILLIAM HENRY, JR.** (C/S—J-M). Field superintendent, Republic Natural Gas Co.

KENTUCKY

Wheelwright—**BANKS, RALPH.** (C/S—S-J). Safety inspector, Inland Steel Co.

LOUISIANA

New Orleans—**SIMMONS, FRED E., JR.** (C/S—J-M). Petroleum engineer, Louisiana Land & Exploration Co.

MASSACHUSETTS

Cambridge—**GRANT, NICHOLAS JOHN.** (C/S—J-M). Associate professor of metallurgy, Massachusetts Institute of Technology.

Southbridge—**STREETER, THOMAS WINTHROP, JR.** (C/S—S-J). Management trainee, American Optical Co.

MICHIGAN

Detroit—**SPENCER, ANDREW R.** (C/S—S-J). Sales engineer, Steel Sales Corp.

East Lansing—**McGRADY, DENTON DELBERT.** (C/S—J-M). Assistant professor of metallurgical engineering, Michigan State College.

Houghton—**WALLACE, ROBERT RUNDLE.** (C/S—S-AM). Junior mining engineer, Oliver Iron Mining Co.

Midland—**SAUNDERS, WILLIAM PUTNAM.** (J). Research and development engineer, Dow Chemical Co.

MINNESOTA

Duluth—**THORPE, DEAN FRANKLIN.** (C/S—S-J). Assistant concentration engineer, Oliver Iron Mining Co.

Hibbing—**HARRISON, HUGH HOWARD.** (C/S—S-J). General manager, Pacific Isle Mining Co.

Nashwauk—**LIESKE, EARL FREDERICK.** (J). Mill draftsman, Design Dept., Cleveland-Cliffs Iron Co.

MONTANA

Butte—**JUDD, KENNETH MOR- TON.** (C/S—S-J).

Libby—**GALE, GEORGE DOUGLAS.** (C/S—J-M). Mineral dressing engineer, Zonolite Co.

MISSISSIPPI

Laurel—**LUBY, MICHAEL ANDREW.** (J). Engineer trainee, Gulf Refining Co.

MISSOURI

Rolla—**KENWORTHY, HEINE.** (C/S—J-M). Metallurgist, U. S. Bureau of Mines.

St. Louis—**BRUNE, ARTHUR WILLIAM.** (C/S—J-M). Instructor in civil engineering, Washington Univ.; **SPHAR, CURTIS WILLIAM.** (C/S—S-J). Mineral Technologist, Missouri Pacific Lines.

NEW JERSEY

Mahwah—**FLINN, RICHARD AL- OYSIUS.** (C/S—J-M). Section head, American Brake Shoe Co.

Maplewood—**NEFF, CHARLES HAROLD.** (M). Geologist, Gulf Oil Co.

Metuchen—**QUADT, RAYMOND A.** (C/S—J-M). Director of aluminum developments, American Smelting & Refining Co.

Morristown—**GETZ, ALBERT J.** (C/S—J-M). Plant engineer, Richard Ore Co.

Summit—**WILLIAMS, RAYMOND T.** (M). Design engineer, Anaconda Copper Mining Co.

NEW MEXICO

Carlsbad—**GORDON, JOHN DAV- IDSON, JR.** (C/S—J-AM). Efficiency engineer, International Minerals & Chemical Corp.

Hanover—**BLAKEMORE, PAGE BLANTON.** (M). Mine superintendent, Peru Mining Co.; **GALASSINI, MARIO.** (M). Mine foreman, Peru Mining Co.; **JEROME, STANLEY EVERETT.** (R, C/S—J-M). Assistant to general superintendent, The New Jersey Zinc Exploration Co., S. W. Dept.

Silver City—**VEEDER ARTHUR KIMBALL.** (C/S—J-M). Manager and chemist, New Mexico Minerals Laboratory.

NEW YORK

Brooklyn—**HILL, KENNETH EVAN.** (C/S—J-M). Petroleum engineer, Chase National Bank.

De Grasse—**WINSLOW, KENELM CRAWFORD.** (C/S—S-J). Engineer, Hanna Coal & Ore Corp.

New York—**FRANK, THOMAS W.** (C/S—J-M). Consultant, Kenmore Metals Corp.; **FRITZ, GLENN H.** (C/S—J-M). Representative, export division, Joy Manufacturing Co.; **HARMON, ROBERT B.** (C/S—S-J). Partner, Harmon Lichtenstein & Co. (representative of Societe de la Vieille Montagne.); **WRIGHT, MYRON ARNOLD.** (C/S—J-M). Executive assistant, Standard Oil Co. (N. J.).

Scarsdale—**READ, THOMAS ALBERT.** (C/S—J-M). Associate professor of metallurgy, Columbia Univ.

Syracuse—**HODAPP, WALTER LEONARD.** (C/S—J-AM). Metallurgist, Crucible Steel Co. of America.

OHIO

Cleveland—**FROST, BENJAMIN BURT.** (C/S—J-M). Sales engineer, Arthur G. McKee & Co.

Elyria—**GOUDVIS, THEODORE L.** (C/S—J-M). President, Concrete Masonry Corp.

Warren—**TOTTEN, PAUL RICH- ARD.** (C/S—S-AM). Plant metal- lurgist, Thomas Steel Co.

OKLAHOMA

Duncan—**KILGORE, JAMES GRO- VER.** (M). Sales manager, Halliburton Oil Well Cementing Co.

Tulsa—**HEMPHILL, JOHN WIL- BORN.** (M). District engineer, Sinclair Oil & Gas Co.; **INGRAM, CHARLES CLARK.** (C/S—J-M). Engineer, Oklahoma Natural Gas Co.; **KRIEDEL, MONROE W.** (C/S—J-M). Research group leader, The Carter Oil Co.; **SINSHEIMER, WARREN A., JR.** (C/S—S-J). Petroleum engineer, Raymond F. Kravis.

OREGON

Coos Bay—**SAMUELSON, RODNEY LEE.** (C/S—S-J). Assistant to general superintendent, head drafts- man, Coast Pacific Lumber Co.

PENNSYLVANIA

Aliquippa—**EDWARDS, ROBERT EUGENE.** (M). Assistant to open hearth and Bessemer superintendent, Jones & Laughlin Steel Corp. **Bethlehem**—**MANCKE, EDGAR B.** (C/S—J-M). Engineer, Research Dept., Bethlehem Steel Co.

Bradford—**VAUGHN, JOSEPH CHARLES.** (C/S—S-J). Petroleum production engineer, Quaker State Oil Refining Corp.

Indiana—**GRAFF, PAUL WILBUR.** (C/S—S-M). President, Westmoreland Mining Co.

Lansford—**GILBERT, JOSEPH C.** (R, C/S—AM-M). Engineer-in- charge, Hazleton Field Station, U. S. Bureau of Mines.

Lebanon—**HORST, RUSSEL JO- SEPH.** (J). Metallurgist, Bethlehem Steel Co.

Narberth—**VAUGHAN, WARNER GLEASON.** (M). Partner, Warner Vaughan Co.

New Kensington—**FITZGERALD, ROBERT DALE.** (C/S—S-J). As- sistant metallographer, Aluminum Company of America.

Philadelphia—**DALLAS, GEORGE MIFFLIN.** (AM). Vice-President, Reading Briquet Co., American Bri- quet Co. & Ecco Manufacturing Co.

Pittsburgh—**SPARR, WILLIAM HENRY.** (C/S—J-M). Metallurgist, The International Nickel Co., Inc.

Pottsville—**JONES, JOHN ED- WARD.** (J). Colliery engineer, Otto Colliery Co.

Scranton—**DONNE, JOHN JEN- KIN.** (M). Division mining engineer, Glen Alden Coal Co.

SOUTH DAKOTA

Lead—**HOFFMAN, FRANCIS GERALD.** (C/S—S-M). Mine sur- veyor, Homestake Mining Co.

TEXAS

Abilene—BRANTLY, MIMS McGEHEE. (J). Drilling engineer, Drilling & Exploration Co., Inc.; BRYSON, FRED EMERSON. (C/S—S-J). West Texas, New Mexico & Colorado district engineering manager, insurance co. inspector, Houston Fire & Casualty Insurance Co. & Companion Cos.; WILSON, HERBERT CLAY (C/S—J-M). Production superintendent, Geochemical Surveys.

Beaumont—MONTAGUE, KENNETH ELWIN. (C/S—J-M). Reservoir engineer, Sun Oil Co.; TUCKER, CHARLES WILLIAM. (R, C/S—S-J). Junior petroleum engineer, Stanolind Oil & Gas Co.

Corpus Christi—GIBSON, ROBERT L. (M). Division reservoir engineer, Shell Oil Co., Inc.; PIERCE, HAROLD FREDERICK. (R, C/S—S-M). Exploitation engineer, Corpus Christi Division, Shell Oil Co., Inc.; RYLANDS, ALBERT THOMAS. (C/S—J-M). Division production manager, Shell Oil Co., Inc.; WATKINS, OWEN MILTON, JR. (C/S—J-M). Production engineer, Pan American Production Co.

Fairbanks—SINEX, BRADFORD JAY. (C/S—J-M). District engineer, Amerada Petroleum Corp.

Falfurrias—CABINISS, BERT ADRON. (M). District manager, Sperry-Sun Well Surveying Co.

Fort Worth—DAGGETT, WILLIS KENNETH. (M). Sales manager, Wellex Jet Services, Inc.

Houston—GLANVILLE, JAMES WILLIAM. (C/S—S-J). Research engineer, Humble Oil & Refining Co.; HOWARD, LEWIS BENTON. (C/S—J-M). Petroleum engineer, American Republics Corp.; NEWSONIE, JOEL ARTHUR, JR. (C/S—J-M). Vice-President, Parks Engineering Co.; WEATHERLY, JUSTIN EUGENE, JR. (C/S—S-J). Petroleum engineer trainee, Gulf Oil Corp.; WINSAUER, WELDON OTTO. (J). Assistant research engineer, Production Research Dept., Humble Oil & Refining Co.

Kilgore—ECKART, ROBERT RONDO, JR. (C/S—J-M). Division production geologist, Shell Oil Co., Inc.

Midland—BONNELL, ROBERT ALFRED, JR. (M). Engineer, Sinclair Oil & Gas Co.; FITTING, ROBERT DANCY. (C/S—J-M). Member of firm, Fitting, Fitting & Jones.; RANDERSON, LUTHER WINN. (C/S—J-M). Petroleum engineer, Magnolia Petroleum Co.; SIMPSON, JULIAN MOORE. (J) Junior engineer, Sinclair Oil & Gas Co.

San Antonio—PARKS, ROBERT HALL. (C/S—S-J). Petroleum engineer, Sunray Oil Corp.; RENFRO, HEATH. (R, C/S—S-M). Production Supt., Ralph E. Fair, Inc.; TOWER, JOHN RUSSELL. (AM). Petroleum engineer, Seeligson Engineering Committee.

Texas City—REIKIE, MATTHEW KER THOMSON. (M). Superintendent of research, Tin Processing Corp.

Wichita Falls—GOULDY, ROLAND. (C/S—J-M). Petroleum engineer, Bridwell Oil Co.

UTAH

Eureka—FITCH, CECIL ALOYSIUS, JR. (C/S—J-M). Assistant general manager, Chief Consolidated Mining Co.

Midvale—TONNESEN, ROBERT SVANE. (C/S—S-J). Mucker, U. S. Smelting & Refining Co.

WASHINGTON

Olympia—RECTOR, MICHAEL ROBERT. (C/S—S-J). Field geologist, Union Oil Co. of California.

Spokane—KAISER, EDWARD PECK. (C/S—J-M). Regional geologist, New Jersey Zinc Co.

WEST VIRGINIA

Holden—NORTHCOTT, ELLIOTT, II. (J). Preparation engineer, Island Creek Coal Co.

Huntington—RUDNICKI, JOHN WALTER FRANCIS. (C/S—S-J). Field engineer, The C. & O. Railway Co.

WISCONSIN

Cudahy—AGUIRRE, LUIS ENRIQUE. (M). Erection engineer, Allis-Chalmers Mfg. Co.

WYOMING

Laramie—FEARN, LYMAN. (M). State inspector of mines, State of Wyoming.

Rock Springs—FISHER, SYLVESTER JAMES. (C/S—S-J). Petroleum engineer, Mountain Fuel Supply Co.

ALBERTA

Calgary—KELLER, ALFRED EMIL. (C/S—J-M). Assistant to manager, Texaco Exploration Co.

BRITISH COLUMBIA

Britannia Beach—CHLUMECKY, NICHOLAS. (AM). Miner, Britannia Mining & Smelting Co., Ltd.; McKICHAN, JOHN DOUGLAS. (C/S—J-M). Ventilation engineer, Britannia Mining & Smelting Co., Ltd.; ROPER, EDWARD CECIL. (C/S—J-M). Manager, Britannia Mining & Smelting Co., Ltd.

ONTARIO

Black Donald Mines—EDWARD, BRUCE GARFIELD. (C/S—J-M). Manager, Black Donald Graphite Division, Frobisher, Ltd.

Ottawa—HAYSLIP, GORDON OSCAR. (C/S—J-M). Mineral dressing engineer, Bureau of Mines.

DURANGO

Tayoltita—WOODS, ROBERT GRAYSON. (C/S—J-M). Mill superintendent, San Luis Mining Co.

GUERRERO

Mexcala—SMITH, WALTER ALTON. (M). Mine superintendent, Cia. Minera del Mexcala.

HIDALGO

Taxco—COOPER, JAMES RENNICK. (C/S—J-M). Metallurgist, American Smelting & Refining Co.

SONORA

Cananea—VELASCO, J. RUBEN. (C/S—J-M). Field geologist, Anaconda Copper Mining Co.

BOLIVIA

Oruro—ALBERTSON, FLOYD ELI. (C/S—J-M). Mill superintendent, Cia. de Mines de Colquiri.

BRAZIL

Rio de Janeiro—DEARAUJO, OLIVEIRA, GABRIEL MAURO. (C/S—J-M). Mining engineer, Dept. Nacional de Producao Mineral.

COLOMBIA

Bogota—COSTER, WILLEM ARNOLD. (C/S—J-M). Consulting engineer, self. HESS, LORAIN. EMMETT. (C/S—J-M). Chief petroleum engineer, Socony-Vacuum Oil Company of Colombia.

ECUADOR

Guayaquil—WILLIAMS, CLARENCE THOMAS. (C/S—J-M). Engineer-foreman in charge of Mines Nuevas operation, Calera Exploration Co.

PERU

La Oroya—LACY, WILLARD CARLETON. (C/S—J-M). Petrologist, Cerro de Pasco Copper Corp.

VENEZUELA

Cumarebo—BOSSERT, WARREN HARRIS. (J). Petroleum engineer, Creole Petroleum Corp.

ENGLAND

Yorkshire—FRANCIS, SYDNEY. (M). Chief metallurgist and managing director, Brontalloy, Ltd.

GERMANY

Stuttgart—SCHRAMM, JACOB. (M). Chief metallurgist, Metall-Guss and Presswerk Heinrich Diehl. Vide; Univ. Teacher, Technische Hochschule.

LUXEMBURG

Luxemburg—SCHMIT, RENE. (M). Superintendent, Aciéries Réunies de Burbach-Eich-Dudelange.

SYRIA

Aleppo—TCHALABI, MOHAMMED TAHER. (J).

INDIA

Bokaro—SHOME, MAKHAN LALL. (M). Superintendent of Collieries, Government of India.

PHILIPPINE ISLANDS

Manila—THORMEYER, WALTER RICHARD. (C/S—J-M). Sales engineer, mining dept., Earnshaws Docks & Honolulu Iron Works, Manila.

STUDENT ASSOCIATES

Elected August 17, 1949

Thomas B. Budinger....Penn State
Noel F. Herbst....Melbourne Univ.
Raghunath C. Deshpande.....RPI
James R. Easterday...Wayne Univ.
Bernhardt H. Petersen
.....Mich.Min. & Tech.
Earl G. Thurman.....Univ. Okla.

Drop Everything...!

in the laboratory



... the pan



... and the rigging



AIME Mid-Year Meeting

Columbus, Ohio, Sept. 24—Oct. 1

Meet: Two thousand engineers

***Learn: Latest operating techniques
at important technical sessions***

Bring: Your experience and "know-how"

Enjoy: Field trips, banquets, social events



Field Trips

ALL day Thursday, September 29 will be devoted to field trips, and Members attending the Mid-Year Meeting can take their choice from among the six outlined here.

1. Battelle Memorial Institute and Ohio State University. Battelle is the largest independent industrial research foundation in the nation, with an international reputation for research in metallurgy,



Flotation Research at Battelle.

fuels, ceramics, corrosion technology and graphic arts. It has gained distinction in fields ranging from industrial physics to engineering economics. At Ohio State, visitors will see a special exhibit by the State's Archaeological and Historical Society, a demonstration of the fire wall tests now being conducted, and the new cyclotron.

2. Hanna Coal Co., Cadiz, Ohio. Coal men and open-pit operators will enjoy this trip through the Company's underground coal mine, as well as the completely modernized coal laundry and coal cleaning plant, where it is now possible to dry coal to within 2 percent of the surface moisture. But the day's feature will be the operation of the world's largest open pit mining shovel, the giant which scoops up 49 cubic yards at one time.

3. Jeffrey Manufacturing Co. and Battelle Memorial Institute. The big 50-acre Jeffrey plant where emphasis is placed on the mechanized aspects of drilling, cutting, loading, conveying, transporting, screening, beneficiation, sizing and ventilation will provide the first half of the trip. Visitors will see mining material handling and processing from raw product to finished mining machine. At Battelle, the accent will be on the research be-

ing carried out by the Bituminous Coal Research Association there.

4. Lustron Corp. and Owens-Corning Fiberglas Co. Members who are bothered by the housing shortage will enjoy the Lustron plant, where 100 steel and enamel dwellings are mass-produced each day. At the Owens-Corning plant in Newark, Ohio, white fleecy blankets of Fiberglas move along on conveyor belts to their ultimate destination as thermal insulation materials in domestic appliances, industrial equipment, and all types of transportation equipment.

5. Pure Oil Refinery and Permanente Metals Corp. This is designed to satisfy both metallurgists and Petroleum Branch Members. Many unusual operations will be available for inspection at the Pure Oil refinery, and the Permanente tour will embrace the Company's operations in working high purity aluminum pig into transmission cable wires. Four dollars for this trip.

6. Turnstead Division, General Motors Corp., and National Electric Coil Co. Plant hardware is produced at the Turnstead Plant for all General Motors automobiles, and visitors will view metal forming, plating, and stamping operations. National Electric Coil manufactures electrical coils to be used in rewinding rotating electrical apparatus.



Wrapping underground pipe with Fiberglas.

The Ladies

Mrs. AIME can relax at the Mid-Year Meeting, and enjoy a round of informal fun. There'll be no long-dress affairs emphasizing formality, including the Mid-Year Banquet at the Neil House.



The Proceedings will start with the registration on **Monday** morning, Sept. 26, and then, at noon, the All-Institute luncheon will begin. That afternoon, the ladies will go on a sightseeing tour of Columbus, through the Governor's mansion, Battelle Memorial Institute, Capital University, Ohio State University, and yes, even the Ohio Penitentiary.

Another inspection trip is scheduled for **Tuesday** morning, followed by luncheon and then all afternoon to get ready for the cocktail party and AIME Mid-Year Banquet later that evening. **Wednesday** will feature luncheon and a fashion show.

Registration will begin Sunday, Sept. 25, at the Neil House, and continue through Monday morning. Members, \$3, non-members, \$5. Members and non-members will be distinguished from one another by different colored registration badges. This will give members an opportunity to extend courtesies to non-members.

Social events: Banquets — AIME Mid-Year Banquet, Wednesday evening. Preceded by a cocktail party. Tickets, \$6.50. Annual Banquet, Southern Ohio Section, National Open Hearth Committee, AIME, on Friday evening. Tickets, \$6. **Others** — Dinner-in-the-Sky, Wednesday evening. Scotch Breakfast, Minerals Beneficiation Division, Tuesday morning.

Luncheons: Monday, All-Institute Luncheon. \$3.50. **Tuesday**, AIME Board of Directors; Extractive Metallurgy Division, (\$3.50); Executive Committee, Coal Division (\$3.50). **Wednesday**, Minerals Beneficiation Division (\$3.50); Coal Division (\$3.50); and Industrial Minerals Division (\$3.50).

Meetings: Missouri School of Mines Alumni will hold a party on Monday evening, Sept. 26. **The Petroleum Branch** will meet on Monday, Tuesday and Wednesday. See the **Journal of Petroleum Technology** for details. **The Mineral Industry Education Division** will meet on Sunday afternoon at the Neil House.



Hugo Johnson

General Chairman, AIME Mid-Year Meeting

Technical Sessions

Mining Geology

Tuesday Afternoon

Technical Session.

Underwater Seismic Investigations for Civil Engineering Studies. By Robert E. Barnett, Geologist, Corps of Engineers, Ohio River Division Laboratories.

Trace-element Studies, Santa Rita, New Mexico. By Paul F. Kerr, Prof., Mineralogy, Columbia University, and Donald L. Graf, Illinois Geological Survey.

Tin Deposit of Monserrat, Bolivia. By Russell Gibson, Assoc. Prof., Geol., Harvard Univ., and F. S. Turneaure, Assoc. Prof., Geol., Univ. Michigan.

Minerals Beneficiation Division

Sunday Afternoon

Informal business session. S. J. Swainson, presiding.

Monday Afternoon

Technical Session, E. H. Rose and Harlowe Hardinge, Chairmen. Junior Ball Room, Neil House.

Unit Process of Crushing and Grinding

Effects of Rod Mill Feed Size Reduction. By John J. Strohl, Mill Supt., and Harry J. Schwellenbach, Mill Operator, National Lead Co.

Use of Spiral Classifiers as Ball Mill Feeders. By T. C. King, Eagle Picher Co.

Relative Wear Rates of Various Diameter Grinding Balls. By

D. E. Norquist, Sheffield Steel Co.

Grinding Progress at Tennessee Copper. By J. F. Myers, Mill Supt., and F. M. Lewis, Asst. Mill Supt., Tennessee Copper and Chemical Co., Copperhill, Tenn.

Tuesday Morning

Scotch breakfast.

Technical Session, E. H. Crabtree, Jr., Chairman. Junior Ball Room, Neil House.

Unit Process of Concentration

The Flotation of Copper-zinc Sulphide Minerals of the Prince Leopold Mine. By Charles Piedboeuf.

Flotation of Chalocite. By S. B. Tuwiner and S. Korman, Dept. of Devel. and Research, Phelps Dodge Corp., Laurel Hill, N. Y.

The Frothability of Pine Oils. By S. C. Sun, Asst. Prof., Mineral Preparation, Penn. State Coll.

The Behavior of Mineral Particles in Electrostatic Separation. By S. C. Sun, J. D. Morgan, Jr., National Security Resources Board, and R. F. Wesner, Engr., McNally Pittsburgh Mfg. Corp.

Tuesday Afternoon

Technical Session, Raymond E. Byler, Chairman.

Unit Process of Operating Control

An Electronic Tramp Iron Detector. By C. M. Marquardt, Mining Engineer and Geophysicist, Combined Metals Reduction Co.

Statistical Control Methods. By W. F. Keyes, Jr., Mineral Engr.,

and M. H. Dorenfeld, Mineral Engr.

Experiences with a Density Recording and Control Instrument for Heavy Media Separation. By J. J. Bean, Min. Dressing Engr., American Cyanamid Co.

Paper on pumping by Wm. B. Stephenson, The Allen Sherman Hoff Co.

Wednesday Morning

Technical Session, I. M. LeBaron, Chairman.

Symposium on Grinding
(titles later)

Wednesday Noon

Minerals Beneficiation Division—Business Luncheon. S. J. Swainson, Division Chairman, presiding.

Wednesday Afternoon

"Clean-up" Session, Donald W. Scott, presiding.

General Discussion of all papers.

Extractive Metallurgy Division

Tuesday Morning

Technical Session.

Titanium Investigations: Research and Development Work on the Preparation of Titanium Chloride and Oxide from Titanium Mattes. By R. G. Knickerbocker, C. H. Gorski, H. Kenworthy and A. G. Starlipper, Met. Div., U. S. Bureau of Mines, Rolla, Mo.

A Thermodynamic Investigation of the System Silver-silver Sulphide. By Terkel Rosenqvist, Institute for the Study of Metals, University of Chicago.

Industrial Minerals Division

Monday Afternoon

Building Materials; Groundwater. Industrial Limestones of Indiana.

By John B. Patton, Indiana Geological Survey.

Limestone in the Muskingum Valley. By C. H. Bowen, Ohio State University Experiment Station.

Raw Material Economics of the Ohio Cement Industry. By R. J. Anderson, Battelle Institute.

Occurrence of Groundwater near Lexington, Kentucky. By D. K. Hamilton, Univ. of Kentucky.

Our Nation's Building Stone. (Film.) By M. J. Morgan, Indiana Limestone Producers Ass'n.

Tuesday Morning

Mineral Deposits.

Silica Deposits of Arkansas. By W. B. Mather, Southwest Research Institute.

Kaolin in Texas. By F. K. Pence, University of Texas.

Mineral Resources of the Lone Star Steel Company. By A. B. Drescher, Lone Star Steel Co.

Geologic Exploration for Uranium in the Colorado Plateau Area. By Wallace G. Fetzer, Atomic Energy Commission.

Tuesday Afternoon

Preparation; New Products.

Problems of Preparation of Limestone Samples for Spectrographic Analysis. By Richard K. Leininger, Indiana Geological Survey.

Petalite—A New Commercial Mineral. By John D. Clarke, Foote Mineral Co.

Sand Deposits of Northern Ohio. By Wm. Smith, Ohio State Univ.

Producing Strong Light-weight Aggregate Using a Wide Variety of Clays. By G. A. Bole and K. B. Czarnecki, Ohio State University Experiment Station.

Refractory Clay Situation in Ohio and Kentucky. By J. O. Everhart, Ohio State University Experiment Station.

Wednesday Morning

Salt Deposits.

Salt Deposits of New York State. By John G. Broughton, New York State Science Service.

Salt in the Northern Appalachian Area. By John A. Ames, Baltimore and Ohio Railroad.

Geology of Michigan Salt Deposits

By Harry J. Hardenberg, Michigan Geol. Survey.

Salt Deposits of Kansas.

Wednesday Afternoon

Wednesday afternoon left open so members of Industrial Minerals Division may attend the Air Pollution session of the Coal Division, at 2 p. m.

Coal Division

Monday Afternoon

Student Forum—joint with all Divisions.

Panel: Moderator, C. E. Lawall, Chesapeake & Ohio Railway Co. A. R. Anderson, Mgr. of Sales, Mining Div., The Jeffrey Mfg. Co., Columbus. C. E. Bales, vice president, Ironton Fire Brick Co. M. D. Cooper, Mgr., Vocational Training, National Coal Association. Gerald C. Gambs, Mgr., Service Dept., Pittsburgh Consolidation Coal Co. James Hyslop, Exec. Vice Pres., Hanna Coal Co. L. F. Reinartz, vice president, Armco Steel Corp. D. T. Ring, vice president, The Preston Oil Company. Clyde E. Williams, director, Battelle Memorial Institute.

Tuesday Morning

Coal Geology and Synthetic Fuels. Coal Reserves of Perry County, Ohio. By Norman K. Flint, Geological Survey of Ohio, Columbus, Ohio.

Research in Coal Geology. By Gilbert H. Cady, Illinois State Geological Survey, Urbana, Ill.

Preparation of Coals for Synthetic Liquid Fuels. By Wm. J. Crentz and J. D. Doherty, U. S. Bureau of Mines, Washington, D. C.

Discussions: Synthetic Liquid Fuel Studies in Ohio. By W. H. Smith, Geological Survey of Ohio, Columbus, and C. A. Bowen, Ohio State University, Columbus.

Tuesday Noon

Luncheon. Coal Division Executive Committee.

Tuesday Afternoon

Symposium on Continuous Mining. The Analysis for a Continuous Mining Machine. By Gerald van Stroh, BCR Mining Development Committee, Huntington, W. Va. **The Colmol, A Continuous Mining Machine.** By Clifford H. Snyder, Sunnyhill Coal Co., Pittsburgh. **The Continuous Miner.** By W. B.

Jamison, Jamison Coal & Coke Co., Greensburg, Pa.

Dosco Continuous Miner.

Wednesday Morning

Promises for A.C. Power Economies with New Mining Methods. By J. R. Guard, Rochester and Pittsburgh Coal Co., Indiana, Pa.

Symposium on Mine Roof Pinning: Latest Developments in Roof Bolting. By Edward Thomas, U. S. Bureau of Mines.

Paper by Lee Siniiff, Consolidation Coal Co. (Ky.), Jenkins, Ky

Mining Methods

Tuesday Morning

Technical Session.

Development Work with Trackless Equipment. By E. A. James, Asst. Gen. Mine Supt., Southeast Missouri, St. Joseph Lead Co.

Wrapping Pillars with Old Hoist Rope. By B. T. Wykoff, Mine Supt., St. Joseph Lead Co.

Stresses About Mine Openings. By Louis A. Panek, U. S. Bureau of Mines, College Park, Md.

Wednesday Morning

Technical Session.

A Classification and Application of Drill Jumbos. By O. J. Neslage and R. W. Jenkins, Joy Mfg. Co. **Pneumatic Sectional Steel vs. Diamond Core Drill Sampling in Brecciated Rock, Aspen, Colorado.** By Paul T. Allsman, U. S. Bureau of Mines, Salt Lake City, Utah.

Development in the Use of Steel for Underground Support. By F. J. Haller, Supt., Mather Mine, The Cleveland-Cliffs Iron Co.

Concrete Slusher Drifts. By Joseph Bernhardt, Mine Supt., Mine No. 4, Bethlehem Steel Co.

Wednesday Noon

Coal Division Luncheon.

Wednesday Afternoon

Symposium on Air Pollution. Ballroom, Neil House.

Some of the Unknown Air Polluting Solids of Alleghany County. By T. C. Wurtz, County of Allegheny, Pittsburgh, Pa.

Air Pollution by Industrial Gases and Liquids. By L. C. McCabe, U. S. Bureau of Mines, Washington, D. C.

Discussions: W. C. L. Hemeon, Industrial Hygiene Foundation, Pittsburgh, Pa. Henry F. Hebley, Pittsburgh Consolidation Coal Co., Pittsburgh, Pa.

Low Pressure Distillation of Zinc from Al-Zn Alloy*

MAX J. SPENDLOVE† and HILLARY W. ST. CLAIR,† Junior Member AIME

Introduction

The problem frequently arises, particularly in refining metals or smelting scrap metals, of separating metals in the metallic state. Many metals may be separated by taking advantage of their difference in vapor pressure. Such separations can be made at atmospheric pressure, but the separations are much more selective and can be carried out at considerably lower temperatures if the distillation is done at pressures of a few millimeters or less in an evacuated enclosure. Until recently, this has not been considered feasible as a metallurgical operation, but the recent improvements that have been made in vacuum technology have broadened the applicability of vacuum processes and have prompted re-examination of low-pressure distillation of metals as a practicable process. The distillation of zinc from lead is one separation that has already been reduced to practice.¹

This paper is the first of a series of studies being made on separation of nonferrous metals by distillation at low pressures. Although these experiments were confined to the separation of zinc from aluminum, the significance of the results is by no means confined to these two metals. The purpose has been to investigate a metallurgical technique rather than merely to devise a means of separating specific metals.

The experimental work on distillation of zinc from zinc-aluminum alloys at reduced pressure grew out of earlier work on distillation at atmospheric pressure.² The earlier work indicated that it would not be practicable to decrease the zinc in the alloy much below 10 pct owing to the high temperature required. Therefore attention was turned to distillation at low pressures, at which lower temperatures are required. After preliminary tests were made in a small, evacuated tube furnace, a larger furnace having a capacity

of 100 to 150 lb of metal per charge was constructed. Distillation tests were first made on pure zinc and then on aluminum-zinc alloys of various composition. Particular attention was given to the limit to which zinc could be reduced in the residual metal. Data were also taken on the rate of evaporation, and heat balances were made for both the crucible and the condenser.

Distillation Furnace

The vacuum-distillation unit is illustrated schematically in Fig 1. The major components are the induction furnace, the condenser, the vacuum system, and the power-conversion unit. Power is supplied to the induction furnace from a 50-kw 3000-cycle motor-driven alternator. The pressure in the furnace is reduced by a vacuum pump having a nominal pumping speed of 10 liters per sec. When in operation, the metal vapors travel upward from the furnace to the water-cooled condenser where they are collected in amounts of 50 to 100 lb. The condenser is removed with aid of an electric hoist. When the system is under vacuum, the condenser is made self-sealing by a rubber gasket between the smooth-faced, water-cooled flanges at the top of the furnace and the bottom of the condenser. The pressure of the atmosphere is more than sufficient to insure sealing. At the conclusion of an experiment, the residual

metal can be removed from the furnace by removing the condenser and tilting the furnace with the electric hoist. The metal was cast into the molds carried on a mold truck. A photograph of the furnace and auxiliary equipment is shown in Fig 2.

The details of the vacuum furnace are illustrated in Fig 3. The furnace proper is made vacuum-tight with rubber gaskets placed at each end of a fused quartz cylinder. A clamping plate at the bottom and a ring at the top are made to squeeze the rubber between the metal and the end of the quartz tube. A large graphite crucible placed inside the quartz cylinder is thermally insulated and physically supported by refractory insulating bricks. A thermocouple in a quartz protection tube is located at the bottom of the crucible; the leads pass through a rubber seal in the bottom plate. The supporting structure for the furnace is an angle iron frame with transite board sides.

The condenser is made in the form of a water jacketed cylinder with an opening to the vacuum line at the top. The bottom has a projecting skirt inside the machined flange to provide additional cooling for the rubber gasket. Condenser sleeves are made in the form of two semicylindrical pieces of sheet metal that fit snugly inside the cooling jacket. The split sleeve facilitates removal of the condensate.

Measurement of Temperature and Pressure

The metal temperature was measured by a platinum-platinum rhodium thermocouple inserted in a well extending up into the bottom of the graphite crucible. During rapid evaporation there is a wide difference in temperature between the surface and the main body of metal in the crucible because of the large amount of heat that must be conducted to the surface to supply the heat of evaporation. The heat of

New York Meeting, February 1950.
TP 2671 D. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before April 1, 1950. Manuscript received March 29, 1949; revision received June 14, 1949.

* Papers by authors of the U. S. Bureau of Mines Staff are not subject to copyright.

† Electrical Engineer and Metallurgist, respectively, College Park Branch, Metallurgical Division, Bureau of Mines, College Park, Md.

¹ References are at the end of the paper.

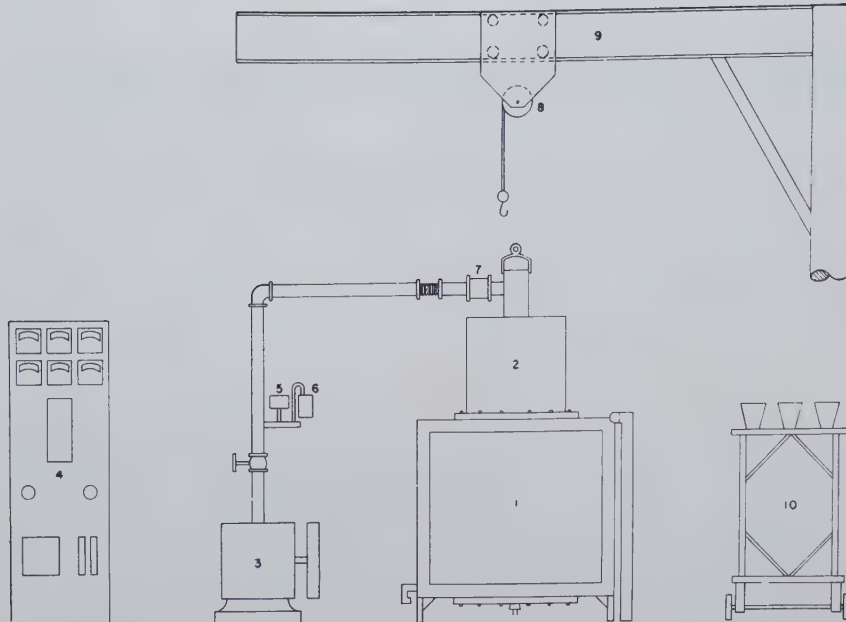


FIG 1—Diagrammatic sketch of equipment for vacuum distillation.

- | | |
|----------------------|------------------------|
| 1. Induction furnace | 6. McLeod gauge |
| 2. Condenser | 7. Dust trap |
| 3. Vacuum pump | 8. Electric hoist |
| 4. Control panel | 9. Mono-rail jib crane |
| 5. Televac | 10. Mold truck |

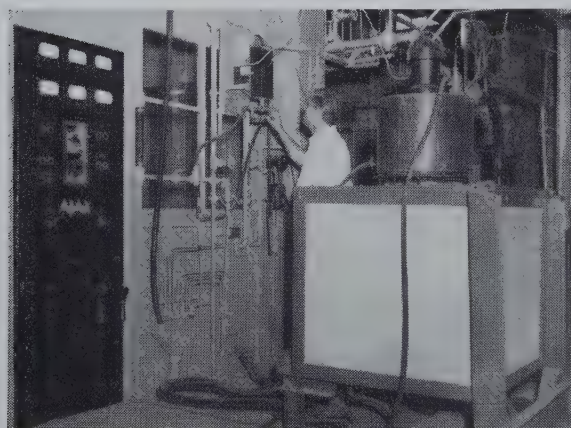


FIG 2—Vacuum distillation furnace.

evaporation amounts to about 200,000 cal per lb of zinc. The temperature gradient required for conducting heat to the surface, neglecting the effect of stirring of the melt due to induced currents in the metal, will be of the order of 10 to 30°C per cm. Therefore, during rapid evaporation, the temperature of the main body of the melt as measured by the thermocouple at the bottom of the crucible may be as much as 100°C higher than the surface temperature. Nevertheless, the temperature of the metal is quite sensitive to changes in the temperature at the surface and gives a fairly accurate representation of relative temperature changes at the surface even though the actual recorded temperatures are considerably higher.

The pressure in the system was measured by a McLeod gauge and by a thermocouple gauge connected to the recording potentiometer. The vacuum indicated by the potential across the thermocouple gauge was checked frequently by the McLeod gauge. Dust filters and cold traps, cooled with dry ice, were placed between the vacuum gauge manifold and the vacuum line. A Pirani gauge was used for leak detection. A small jet of helium was passed over the walls where leaks might occur. When the stream of helium impinged against the leak, the Pirani gauge would indicate a sudden rise in pressure.

The pressure indicated by the vacuum gauge is the partial pressure of the residual gas in the system. There

is no way of directly measuring the partial pressure of the zinc vapor. During evaporation the partial pressure of the residual gas in the region between the metal and the condenser is much lower than is indicated by the vacuum gauge because the zinc vapor tends to sweep the residual gases toward the condenser. In other words, the zinc vapor acts as a diffusion pump, removing gases from the crucible and discharging them at the condenser. Therefore, during rapid evaporation the vapor above the metal is virtually all zinc vapor. The pressure of the residual gases indicated by the vacuum gauges applies only to the condenser and the lines to the vacuum pump.

Experimental Procedure

Weighed amounts of zinc or zinc and aluminum were melted together in the carbon crucible preceding the test. After the metals were thoroughly melted and blended, a sample was taken for chemical analysis. The condenser and baffle were put in place and the metal was allowed to freeze before the system was evacuated. If this precaution was not taken, the rapid evolution of gas from the molten metal during initial stages of evacuation sometimes caused spattering of the alloy into the condenser. After the system was evacuated, the metal was remelted, and the power was increased to the desired level. The flow of cooling water to the condenser was regulated during the test to keep the temperature of the water leaving the condenser less than about 90°C. The flow of water was measured by a rotameter. At the conclusion of a test the condenser was removed from the furnace and allowed to cool. Samples were taken from the condensate and the residual metal for chemical analysis and the condensate and residual metal were weighed.

Experiments 1 to 6 were made with charges of pure zinc. The purpose of these tests was to remedy any operating difficulties before introducing the aluminum and to obtain data on pure zinc for comparison with those to be obtained on zinc-aluminum alloys.

After the preliminary tests, cylindrical sleeves were placed in the condensing chamber to permit easy removal of the condensate. These sleeves were painted before each test with a thin paste of lampblack and water to prevent the condensate from adhering to the sleeve. The paste was allowed

to dry before placing the sleeves in the furnace.

Radiation baffles were placed between the crucible and condenser to prevent direct radiation of heat to the condenser and to equalize the distribution of condensate. Three types of baffles were tried as shown in Fig 4. The baffles were constructed of iron and painted before each experiment with lampblack to protect the iron from the hot zinc vapor.

Experiment 1 was run without the radiation baffle. The deposit was clean and fairly dense but showed evidence of refluxing. The condensate from experiment 3, with a single-deck baffle in place, was cleaner, denser, and showed no evidence of refluxing. Improvements in the condensing sleeve were worked out in experiments 3 and 4. In these experiments the single-deck baffle directed most of the vapors to the lower portion of the condenser, thereby constricting the path of the vapor as the deposit grew out from the walls. In experiment 5 the baffle was inverted to direct the flow of vapor higher into the condenser. This change overcorrected the distribution, resulting in excessive condensation at the top of the condenser and clogging the exit to the vacuum line. In experiment 6, a two-deck baffle was made and placed within the condenser as shown in Fig 4. This baffle gave a fairly uniform deposit. Although baffles were useful in this furnace, it is doubtful that they are absolutely necessary. The same effects doubtless could be reached by properly proportioning the diameters of the upper part of the crucible and the condenser.

The metal temperature provides a good indication of what is happening in the distillation furnace. As the temperature reaches the melting point, an arrest is indicated on the temperature chart. The temperature climbs steeply until it reaches the point at which evaporation begins; then, as the heat supplied is gradually offset by the heat required for evaporation, the temperature levels and remains nearly constant for the remainder of the test. The beginning of distillation is also marked by a sudden rise in the temperature of the cooling water from the condenser as heat is transferred to the condenser by the zinc vapor. A typical temperature curve for the metal temperature is shown in Fig 5, together with a corresponding curve showing the variation of temperature of the cooling water from the condenser. It will be ob-

served that the temperature rises linearly after melting, as shown by section *AB*. The beginning of evaporation is indicated by *C*, the point at which the temperature curve begins to break away from *AB*. It will also be observed that the temperature of the cooling water starts to rise at the melting point and then begins to rise abruptly as the temperature reaches *C*. The rate of evaporation in each experiment was calculated, using point *C* as a starting point.

Distillation Tests on Pure Zinc

Six tests were made on distillation of pure zinc (99.99 pct Zn); the data for these tests are summarized in Table 1.

Evaporation began when the metal temperature reached about 470°C. The maximum metal temperature during evaporation was determined chiefly by the power input; for an input of 10 kw, the temperature leveled at about 500°C and for 30 kw at about 550°C. The metal temperature was also influenced by the distance

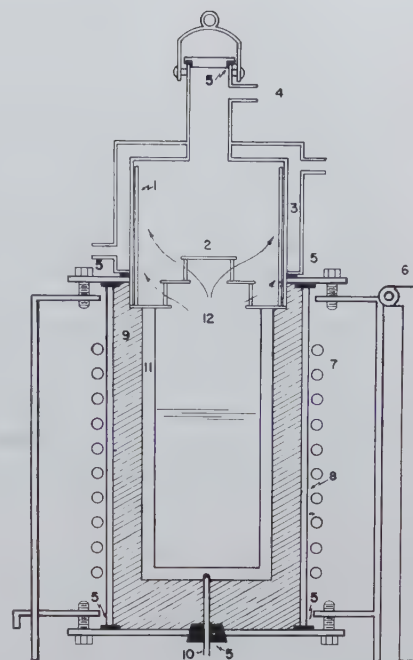
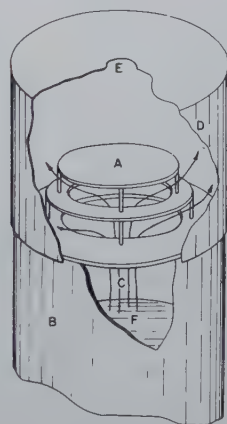
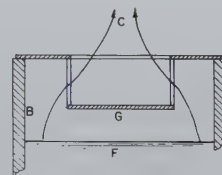


FIG 3—Cross-section of 50 kw distillation furnace.

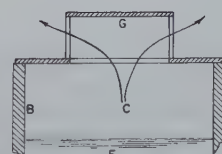
- | | |
|---------------------------|------------------------------|
| 1. Split condenser sleeve | 7. Induction coil |
| 2. Radiation baffle | 8. Quartz tube |
| 3. Water jacket | 9. Insulation |
| 4. To vacuum pump | 10. Quartz thermocouple well |
| 5. Rubber seal | 11. Graphite crucible |
| 6. Furnace pivot | 12. Vapor path |



DOUBLE-DECK Baffle ARRANGEMENT



INVERTED Baffle



UPRIGHT Baffle

FIG 4—Arrangement of baffles between crucible and condenser.

- | | |
|-----------------------|-----------------------|
| A. Double-deck baffle | E. Vacuum take-off |
| B. Carbon crucible | F. Metal surface |
| C. Zinc vapor | G. Single-deck baffle |
| D. Condenser | |

between the evaporation and condensing surfaces and by an obstruction to the flow of vapor to the condenser. This effect is shown by the temperature curves for tests 3, 4, 5, and 6 shown in Fig 6. Tests 3 and 4 were run with the single-deck baffle. This baffle constricted the flow of the vapor as it left the crucible and directed it toward the walls of the condenser. Most of the zinc condensed as large clusters of

crystals in the lower part of the condenser. In test 5 the baffle was inverted as shown in Fig 4, thereby constricting the flow even more than in tests 3 and 4 and directing the vapor to the top of the condenser. This caused the temperature to rise to 540°. The double-deck baffle used in test 6 offered much less impedance to the vapor flow and caused more uniform distribution of the deposit. This ex-

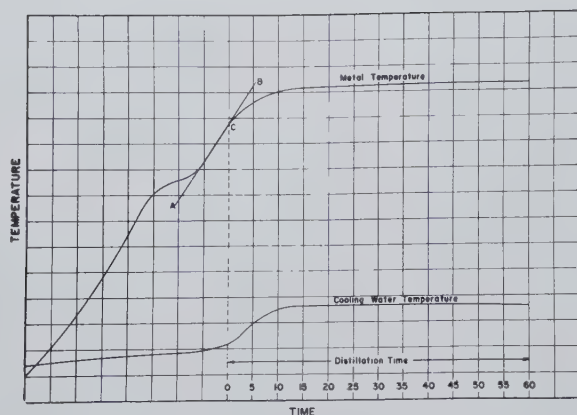


FIG 5—Furnace temperature and cooling water temperature during typical distillation test.

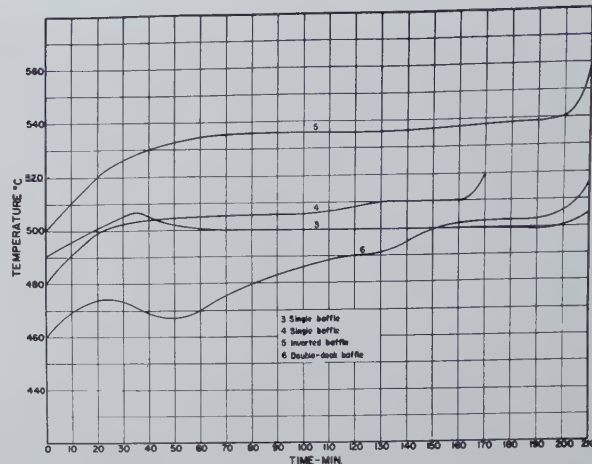


FIG 6—Metal temperatures for tests 3 to 6 showing effect of baffles.

Table 1 . . . Distillation Tests on Molten Zinc

Number	1	2	3	4	5	6
Power input, kw	27.5	25	10	10	10	10
Duration, min	16.5	60	220	115	215	230
Zn distilled, lb	9.8	33	58	41	50	61
Metal temp., avg. °C at which evap. starts	545	536	500	507	533	486
Final pressure, mm Hg	0.700	4.30	0.050	0.120	0.100	0.060
Rate of evaporation, lb per min	0.594	0.550	0.264	0.264	0.232	0.264
Power consumption kw-hr per lb	0.772	0.758	0.622	0.630	0.716	0.629

plains the low average metal temperature during test 6. The temperature first leveled slightly above 470°; during the early part of the test the vapor traveled only a very short distance before it was condensed. As the zinc gradually condensed higher on the walls of the condenser where cooling was more effective, the temperature of the metal gradually rose to 500°C, the same as for test 3.

Referring to Table 1, it will be noted that the rate of evaporation is related more directly to the power input than to the metal temperature. This is not surprising since the rate of evaporation is determined by the rate at which heat is conducted to the surface of the metal; the metal temperature automatically adjusts itself to bring the two in balance. The relationship between rate of evaporation and metal temperature will be discussed again later.

Distillation Tests on Zinc-aluminum

Six distillation tests were made on zinc-aluminum alloy containing about 80 pct zinc. The alloy was made up of commercially pure aluminum (99.89 pct Al) and pure zinc. The zinc and the aluminum were melted and heated

until the aluminum was dissolved. The melt was then skimmed thoroughly before the condenser was placed on top of the furnace. In the first series of tests, only 60 to 80 pct of the zinc was distilled. Tests were made with the power input adjusted to 10, 20, 30 kw, respectively. Temperature of the metal, pressure of the system, and temperature of the cooling water of the condenser were all recorded automatically on a strip-chart potentiometer. A photograph of the chart for test 9 is shown in Fig 7. The chart is calibrated to read the furnace temperature directly in hundreds of degrees centigrade. The pressure and water temperature are recorded on the same chart in arbitrary units. Several actual values for the water temperature and pressure are shown on the chart. The horizontal lines indicate ten minute intervals.

In this test it will be observed that the metal temperature, which had been raised to about 700°C to dissolve the aluminum, dropped abruptly as soon as the vacuum pump was started. The rapid cooling of the metal is a result of rapid evaporation. When the power was turned on the metal gave off dissolved gases and the pressure in the system rose from 80 to 130 microns, then gradually decreased to about 90 microns as the metal was degassed.

The water-temperature variations

were a result of manual adjustment of the flow of cooling water. It was intended that the water should remain at 80°C but the valve adjustment was too coarse to permit accurate control during this test.

The undulations in the metal temperature are interesting. They were always observed when the temperature of the metal was in the liquidus-solidus range. They are explained as follows: As evaporation continues, the zinc in the melt is decreased until the metal temperature is equal to or perhaps less than the liquidus temperature. Then the solid phase crystallizes out; and since it is lighter than the melt, it floats on top. This forms a protective layer on the surface and decreases the rate of evaporation, allowing the temperature to rise until the solid phase is redissolved. Then the same sequence starts all over again, producing a series of undulations in the temperature. It will be noted that simultaneous variations occur in the pressure, indicating that gas evolution increases and decreases with the temperature throughout the test.

The temperature of the melt gradually increases as the proportion of zinc decreases. The vapor pressure of zinc in zinc-aluminum alloys shows a positive deviation from Raoult's law; that is, the vapor pressure is greater than that calculated by multiplying the vapor pressure of zinc by the molal fraction of zinc in the melt. The vapor pressure of zinc-aluminum alloys was measured by Schneider and Stoll³ at 650 to 800°C. They found that their data were in good agreement with Hildebrand's⁴ equation for activity of a solute, in which the coefficient β equals 0.50 or

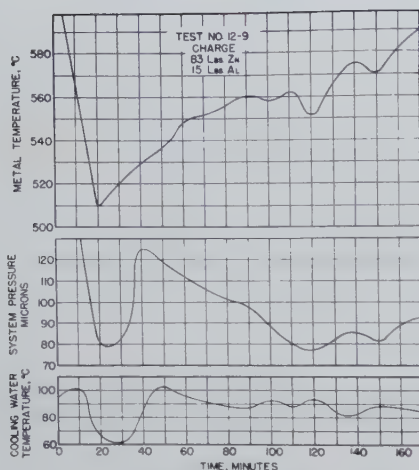


FIG 7—Potentiometer chart for test 9 showing metal temperature, pressure in system, and temperature of water from condenser.

$$\log \lambda_{Zn} = \log \frac{p}{x_{Zn} p_0} = 0.50 (x_{Al})^2$$

where p_0 is the vapor pressure of zinc; x_{Zn} and x_{Al} are the respective molal fractions. From this equation and the vapor pressure of pure zinc it is possible to calculate the vapor pressure of molten zinc-aluminum alloys at any temperature and composition.

Evaporation of pure zinc begins when the vapor pressure reaches about 0.5 mm as indicated by the metal temperature. This would mean that evaporation begins at about 470°C for pure zinc, 560° for 10 pct Zn, 690° for 1 pct Zn, and 800° for 0.1 pct Zn.

If we carry this assumption farther it might be expected that the temperature of the surface of the metal would vary in such a way as to maintain a constant vapor pressure with changing composition. In that case, the temperature at constant power input would vary as in Fig 8, in which are shown the isobaric temperature curves for zinc-aluminum melts having partial pressures of 0.5 mm and 1.0 mm of mercury. However, it was observed, as shown by the temperature curves in Fig 9, that the temperature actually increased more than would be indicated by the isobaric curves of Fig 8. For example, the temperature of test 8 at a power input of 10 kw should have increased 15 to 20°C, according to the change in composition of the residual metal, but the observed increase in temperature shown in Fig 9 was 70°C. The discrepancy for the other two tests at higher heating rates is even greater. As we have already pointed out, the metal temperature may differ appreciably from the surface temperature

during rapid evaporation; but even after taking this into account, it is apparent that the isobaric temperature curves do not provide an adequate guide for predicting the rise in metal temperature.

Another factor to be considered is the nonuniformity of composition arising from rapid evaporation of the zinc at the surface. Zinc will tend to evaporate faster than aluminum can diffuse away from the surface, thereby causing a surface enrichment in aluminum. Accordingly, the observed temperature rise could be explained if the actual surface concentration of zinc in test 8 were between 10 and 20 pct zinc instead of 70 pct as given by the average composition. Likewise, an even greater concentration of aluminum at the surface would be expected at the higher heating rates in tests 11 and 14. The temperature rise in test 14 would indicate only about 2 pct zinc at the surface at the end of the test.

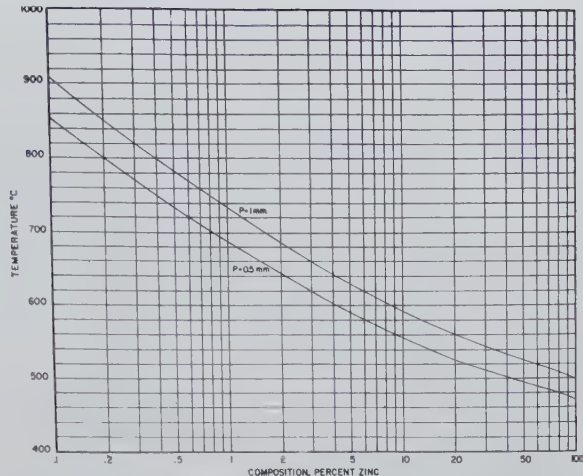


FIG 8—Temperature-composition curves for constant partial pressure of zinc over zinc-aluminum melts.

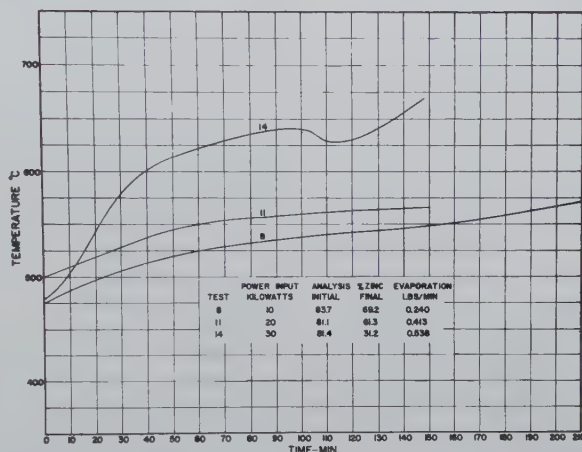


FIG 9—Variation of metal temperature during distillation of zinc from zinc-aluminum melts.

Extent of Removal of Zinc from Aluminum

A series of three tests was run in which the zinc in the alloy was reduced in successive steps. The progressive rise in metal temperature during these tests is shown in Fig 10. The power input to the furnace during the three tests was held at 10 kw. During the first test the metal temperature rose to 512° and remained constant to the end of the test; about 60 pct of the zinc was evaporated, leaving residue containing 34.5 pct zinc. In the second test, about 35 pct more of the zinc was distilled. The metal temperature during this time rose from 512 to 620°. During the final test the temperature rose continuously to more than 900°C and the zinc in the metal was reduced to 0.21 pct.

Comparative tests were made on alloys prepared from aluminum and from aluminum scrap to determine the effect of impurities and other metals

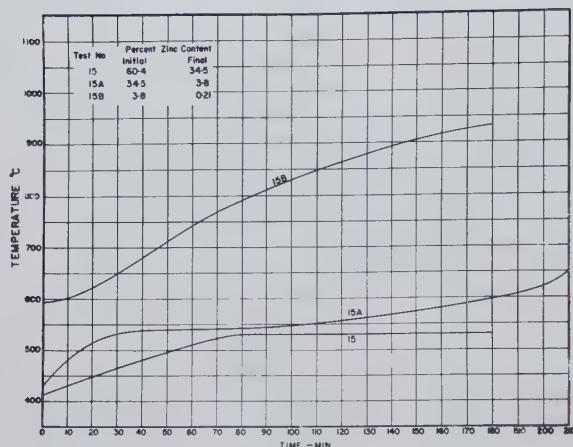


FIG 10—Metal temperature during three successive distillation tests on which zinc was reduced from 60 to 0.2 pct.

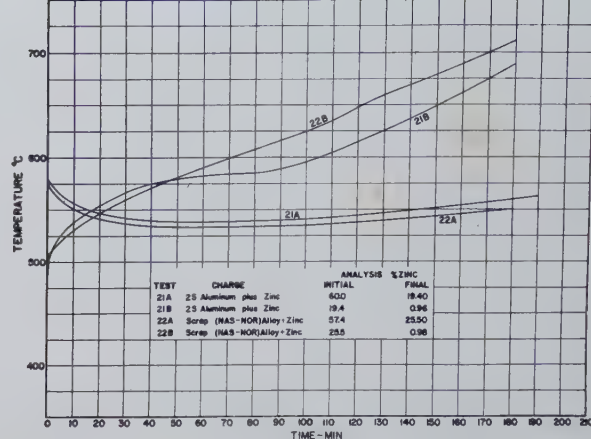


FIG 11—Metal temperature curves for comparative tests with pure aluminum and aluminum scrap.

Table 2 . . . Summary of Distillation Tests on Zinc-Aluminum Alloys

Test Number	8	9	10	11	12	14	15	15A	15B	21A	21B	22A	22B
Power input, kw.	10	20	20	20	30	30	10	10	10	10	10	10	10
Temperature, °C													
Final.	575	590	550	570	555	630	532	610	930	565	685	550	710
Average.	546	557	554	553	555	605	504	558	771	549	594	543	613
Zn content, Pct													
Initial.	83.7	55.8	75.2	81.1	81.4	81.4	60.4	34.5	3.8	60.0	19.4	57.4	25.5
Final.	69.2	23.6	66.3	61.3	77.1	31.2	34.5	3.8	0.21	19.4	0.96	25.5	0.98
Rate of distillation, lb per min.	0.240	0.441		0.413		0.538	0.205	100		0.187	0.123	0.215	0.103
Power consumption, kw-hr per lb.	0.695	0.811		0.808		0.930	0.814	1.67		0.892	1.35	0.776	1.62

present in the scrap in minor amounts. The scrap aluminum came from melted aircraft scrap from the Naval Air Station at Norfolk, Virginia. It was designated by the symbol, "NAS-NOR." A representative analysis is given as follows:

Analysis of NAS-NOR Alloy in Percent	
Aluminum	93.88
Zinc	1.09
Iron	0.91
Silicon	0.31
Copper	3.81

Tests 21A and 21B (Fig 11) were run with 2S aluminum and tests 22A and 22B, with NAS-NOR alloy. The original alloy for each test was made up to about 60 pct zinc. Each test was interrupted after 3 hr and a sample was taken of the residual metal. Then evaporation was continued for another 3 hr, during which the zinc in the residual metal was decreased to less than 1 pct. There was no appreciable difference in the metal temperatures during the first 3-hr period but during the last 2 hr the metal containing the impurities was about 30° hotter. The over-all rates of evaporation were very nearly the same, although more zinc was distilled in the first half of the test with scrap aluminum. All in all, there was no significant difference in the general behavior of the two alloys during distillation.

The important data for all the tests

on zinc-aluminum alloys are summarized in Table 2.

Nature of Condensate

The lower section of the condensate consists of large, dendritic clusters of crystals projecting outward from the walls and in a direction opposite that of the vapor flow. The deposit in the upper part of the condenser is thinner, more compact, and the crystals are much finer. This is illustrated by the photograph in Fig. 12. The form of the crystals in the crystal aggregates is greatly influenced by the rate of condensation and the partial pressure of the permanent gas in the system. At low evaporation rates and low pressures, the condensate is composed largely of fine, loosely compacted, needle-shaped crystals. At higher rates of condensation, the crystal aggregates become more massive and compact and at very high rates the deposit becomes compact and the surface becomes smooth and rounded as though the zinc were deposited in the molten state. The same effects are observed when the pressure is increased, except that the surface takes on a dull appearance instead of the bright, metallic surface produced at low pressures. The dull appearance may result in part from some surface oxidation at higher pressures.

The principal impurities found in the condensate were aluminum, iron, and cadmium. The iron is usually less than 0.02 pct; it was probably introduced as a result of contact with the condenser walls. The cadmium was introduced as a minor impurity in the zinc. It was present only in trace amounts. Aluminum is the only impurity that is inherently important. The vapor pressure of aluminum is only about 10^{-9} times that of zinc at 500°C, but when an alloy of aluminum and zinc is distilled the temperature increases and the partial pressure of the zinc decreases thereby increasing the proportion of aluminum in the vapor. For example, the aluminum in the vapor from a 10 pct zinc alloy at 600° is less than 10^{-4} pct. At 900° the vapor pressures of zinc and aluminum are 700 mm and 1.3×10^{-3} mm, respectively. The vapor pressure of zinc over a 0.1-pct zinc alloy is calculated as 0.91 mm, so that the condensate from such an alloy would contain aluminum and zinc in the proportions to give a deposit containing 0.14 pct aluminum.

The average analyses of the condensates obtained in these tests range from less than 0.01 to about 0.5 pct aluminum. The analyses of the condensates and residual metal for tests 15, 21, and 22, the three series in which the zinc in the residual metal was

reduced below 1 pct, are listed as shown below. The analyses for tests 21B and 22B show that the aluminum in the condensate increases markedly as the zinc is depleted in the residual metal. Furthermore, the aluminum content

Test Number	15	15A	15B	21A	21B	22A	22B
Residual metal, pct Zn.....	34.5	3.8	0.21	19.4	0.96	25.5	0.98
Condensate, pct Al.....	0.20	0.10		0.04	0.34	0.05	0.56

of the condensate greatly exceeds that which can be explained on the basis of evaporation of aluminum, indicating that the aluminum in the condensate is largely a result of spattering.

Spattering seems to occur chiefly during degassing of the molten metal. As the dissolved gas is expelled it carries with it a fine spray of metal formed when the bubbles break at the surface. Spattering from this cause can be reduced considerably by heating the metal very slowly during the time gases are being released. By following such a procedure, it was possible to reduce the aluminum in the condensate to less than 0.01 pct. However, when the zinc in the residual metal is reduced to a few tenths percent, some spattering seems to occur even when there is no evidence of gas evolution. This might be explained on the basis of the concentration of aluminum at the surface during a rapid evaporation. Normally vapor is formed only at the surface because the vapor pressure of the zinc is so low that the hydrostatic pressure even slightly below the surface is too great to permit the formation of bubbles. But as the surface layer becomes depleted in zinc, the temperature will rise to a point where the vapor pressure in the metal immediately under the surface layer will suffice to offset the slight hydrostatic pressure thereby allowing bubbles of zinc vapor to form just below surface. The breaking of these very small bubbles will cause a fine spray of metal some of which may be mechanically entrained in the zinc vapor and carried to the condenser. These droplets must be very minute to be carried by the attenuated zinc vapor, so it is likely that they would be extremely difficult to separate from the vapor. Therefore the condensate probably will contain several tenths of 1 pct aluminum when the zinc in the residual metal is reduced below 1 pct, even though elaborate precautions are taken to avoid contamination by spattering.

Heat Requirements

After distillation has reached a constant rate, the heat requirements consist of the heat of vaporization at the distilling temperature and the

heat necessary to supply losses by radiation and conduction. The heat required for vaporization is transferred by the vapor to the condenser and absorbed by the condenser cooling water. The remainder of the heat input is lost by radiation and conduction from the walls of the furnace and is approximately equivalent to the heat required merely to maintain the furnace temperature.

Tables 3 and 4 give the heat balances for the furnace and the condenser, respectively, for the six tests on distillation of pure zinc. Table 3 was determined by balancing the electrical power input to the furnace, after correcting for the losses in the electrical circuit, against heat removed by radiation and evaporation. The heat losses of the furnace by radiation and conduction were measured by a preliminary test in which the power requirements were determined for maintaining the furnace at various

Table 4 . . . Heat Balance for Condenser

Test No.	Heat of Condensation Kg-Cal per Min.	Heat Abstracted by Cooling Water Kg-Cal per Min.	Heat Not Accounted for Kg-Cal per Min.
1	128.10	181.44	-106.75
2	118.60	164.4	-45.80
3	65.80	86.39	-20.59
4	58.80	61.18	-2.38
5	51.35	69.14	-17.79
6	57.15	60.30	-3.15

Heat of Condensation = ΔH_v for Zn_g → Zn_l at 100°C
= 215.76 kg-cal per lb

temperatures when no distillation was taking place.

Evaporation of Metals at Low Pressure

Evaporation of liquids at low pressure is in many respects quite different from evaporation at atmospheric pressure. At low pressures the atmosphere above the liquid is made up almost entirely of molecules of vapor from the liquid. Vapor forms only at the surface and not in the body of the liquid such as occurs when volatile liquids boil at atmospheric pressure. Evaporation takes place at a rapid rate at low temperatures because the diffusion of vapor molecules away from the surface is not retarded by the presence of other gas molecules. The acceleration of evaporation by reduction in pressure is frequently explained

Table 3 . . . Heat Balance for Distillation Furnace

Test No.	Power Input Kw	Temp. °C	Rate of Evaporation, Lb per Min.	Heat Input* Kg-Cal per Min.	Heat of Vaporization Kg-Cal per Min.	Heat Loss Rad., Cond. Kg-Cal per Min.	Losses Not Accounted for Kg-Cal per Min.
1	27.5	545	0.594	362.5	117.8	87.5	157.2
2	25	536	0.550	329.5	109.1	85.0	135.4
3	10	500	0.305	131.8	60.8	76.2	-5.2
4	10	507	0.273	131.8	54.3	77.5	-0.0
5	10	533	0.238	131.8	47.2	84.2	+0.4
6	10	486	0.265	131.8	52.9	72.8	6.1

* Calculated on basis of average electrical efficiency of 92 pct applied to furnace input.

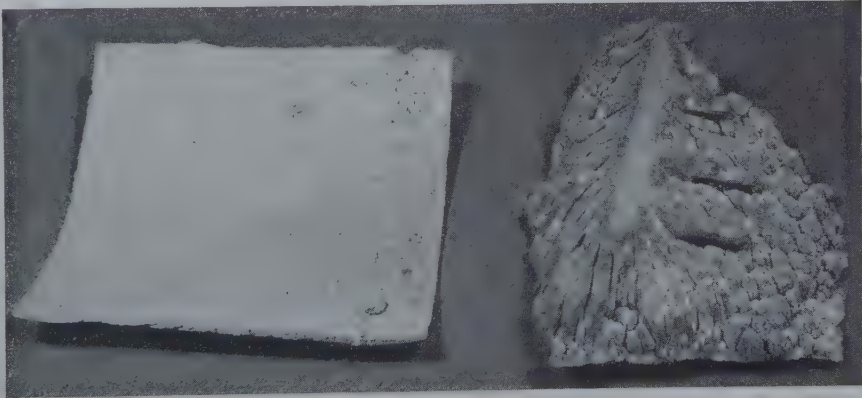


FIG 12—Zinc condensate showing large dendritic clusters found near bottom of deposit and dense fine grained deposit from upper wall.

as a result of the reduction in boiling point. This explanation applies as long as the total pressure remains appreciably greater than the partial pressure of the liquid, the boiling point being defined as the temperature at which the vapor pressure of the liquid is equal to the pressure of the permanent gas in the system. However, at very low pressures the rate of evaporation is limited not by the gas molecules but by the vapor molecules themselves. Under such circumstances the vapor pressure immediately above the liquid exceeds the partial pressure of the permanent gas and the boiling point no longer has any meaning.

The rate at which molecules of vapor leave the surface of the liquid is given by the effusion formula:

$$\omega_0 = p_0 \sqrt{\frac{M}{2\pi RT}} \quad [1]$$

where ω = rate of evaporation in grams per sq cm per sec.;

p_0 = vapor pressure of liquid in dynes per sq cm

M = molecular weight

R = gas constant in ergs per mol per °C

T = temperature °K

Expressing the rate of evaporation in grams per minute, the rate of evaporation of zinc is given by

$$\omega = 28.29 \frac{p_0}{T^{1/2}} \text{ g per sq cm per min.} \quad [2]$$

This equation gives the rate at which zinc would be evaporated into a vacuum or when evaporation is taking place at a comparatively slow rate. However, at high rates of evaporation the molecules of zinc leaving the surface interfere with each other and some of them return to the liquid. The effusion formula [1] may also be used to express the rate at which zinc is being recondensed on the surface:

$$\omega' = p \sqrt{\frac{M}{2\pi RT}} \quad [3]$$

where p now equals the partial pressure of the zinc vapor in contact with the surface. Therefore the net rate of evaporation is given by the difference between ω_0 and ω' or

$$\omega = (p_0 - p) \sqrt{\frac{M}{2\pi RT}} \quad [4]$$

For zinc, this becomes

$$\omega = \frac{28.29}{T^{1/2}} (p_0 - p). \quad [5]$$

When the rate of evaporation reaches a steady state the net rate of evapora-

tion as given by Eq 5 will be equivalent to the rate at which heat is transferred to the surface. The heat required to evaporate 1 g of zinc is 439 cal per g at 500°C. The temperature gradient required to conduct heat to the surface for evaporating at the rate of 1 g per sq cm per min. is equal to 53°C per cm. Therefore, the surface temperature at high rates of evaporation is a good deal lower than the temperature of the main body of metal.

The net rate of evaporation must also be equal to the rate at which vapor is diffused away from the surface. The rate of diffusion is determined by the pressure gradient of the zinc vapor between the evaporating and condensing surfaces. This explains why the temperature of the metal during evaporation is so responsive to obstructions between the crucible and condenser or changes in the distance between the evaporating and condensing surfaces. Any increase in the length of path of the vapor flow or in the pressure of the zinc at the condenser surface produces a corresponding increase in p , the pressure of the zinc at the evaporating surface. This requires a corresponding increase in the vapor pressure, p_0 , and the metal temperature as defined by Eq 5.

Another interesting effect to be considered in connection with the flow of zinc vapor to the condenser is the adiabatic cooling it undergoes. The temperature of the vapor in expanding from pressure p_1 at the crucible to pressure p_2 at the condenser decreases in the ratio

$$\frac{T_1}{T_2} = \left(\frac{p_0}{p_2}\right)^{3/2} \quad [6]$$

The pressure of the zinc vapor at the evaporating surface is considerably less than the vapor pressure of zinc at the surface temperature or its temperature is above the dew point. However, as it is cooled adiabatically, the vapor becomes saturated so that even before it reaches the condenser, the temperature may be below the saturation temperature so that the condensation takes place from a supercooled vapor. For example, if the pressure of zinc vapor at the surface is 0.20 mm when the surface temperature is 500°C, the vapor at this point is only 20 pct saturated. However, when the vapor has expanded to a pressure of 0.15 mm the temperature will have dropped to the melting point, at which the vapor pressure of zinc is only 0.12 mm, and so the vapor is supersaturated with respect to liquid zinc. If the initial

pressure were a little lower the vapor would become saturated at a temperature below the melting point and would be supersaturated with respect to solid zinc.

This provides an explanation of the marked change in appearance that is found in the condensate at different rates of evaporation. At low rates the condensation takes place from a vapor that is supercooled with respect to the solid phase so the vapor condenses directly as a crystalline solid. At higher rates of evaporation, the vapor condenses from a vapor that is supercooled with respect to the liquid phase, so the vapor condenses momentarily as a liquid, chilling immediately to a solid. This deposit is more dense and compact and explains the rounded surfaces that give the appearance of having been melted.

These interesting aspects of low-pressure distillation of metal are discussed briefly to give a qualitative picture of the interrelation of the various factors that determine the rate of evaporation and the nature of the condensate. A more detailed study is being made of these phenomena which will be given a more extensive discussion in a later paper.

Acknowledgment

This investigation has been made by the Metals Recovery and Refining Section at the Eastern Experiment Station of the Bureau of Mines, College Park, Maryland. Acknowledgment is made to L. H. Banning and H. Bizot, formerly members of this group, who participated in the early experiments. Special acknowledgment is made to George E. Biser for his able assistance in constructing and operating the furnace; to A. M. Sherwood for the chemical analyses; to M. J. Peterson for spectrographic analyses; and to other members of the Eastern Experiment Station who contributed to this work.

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Determination of Boundary Stress during the Compression of Cylindrical Powder Compacts

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Introduction

At the present time, the designer of dies for metal powder pressing is handicapped by relative ignorance of stress distribution and frictional effects at the interior surface of the die. Unckel¹ was the first to develop a method for the study of wall friction. He used three Brinell balls on which the die rested during pressing. The total frictional wall force was determined by the size of impression these balls left on a soft metal plate. Since the method does not give radial pressures, or distribution of such pressures, coefficients of friction could not be determined. Although Unckel measured density distribution, he was not able to determine radial or shear stresses. Shaler² has proposed theoretical expressions for the stress and density distribution within cylindrical compacts during pressing, in accordance with the experimental results of Kamm, Steinberg, and Wulff.³

By application of Siebel's method,⁴ Kamm et al³ plotted stress trajectories for two compacts. From the stress trajectories they calculated coefficients of friction from point to point along the die wall. As pointed out by Shaler in the discussion of Ref. 3, these values are based on progressive point-to-point calculations on finite size grid squares across the compact. In the region of the die wall such calculated values may therefore have considerable cumulative error.

The purpose of the present paper is to develop an experimental method by which the nonhydrostatic pressures and shears acting on the interior wall of a cylindrical die can be measured. Such measurements can then be correlated with existing data to aid in the explanation of the pressing process.

The method used is based on the elastic properties of the thick-walled

tube used as the die. The principle of super-position of force systems on an elastic body is assumed to hold. Electric strain gauges were mounted in adjacent positions on the exterior die wall in order to get an exact measurement of the variation of tangential strain over the length of the die during pressing. While in this paper, measurements in terms of only tangential strains are considered, it is well to note that similar calculations may be set up for axial strains. The latter are not preferred, since they tend to be smaller than the tangential strains and therefore permit less sensitive measurements. Discussion in this work is restricted to compacts pressed from both ends, since the elastic deformation of the die is then more amenable to analysis.

Before choosing the electric strain gauge method, a more direct line of attack was considered and discarded. The discarded idea was the insertion of a pressure gauge through a hole in the die wall.* The gauge would have been in the form of a small piston. If pressure were exerted against such a gauge, it would move outward along a radius of the die. One disadvantage of the scheme is its inability to measure shears along the die wall. Another

* Since the completion of this paper a manuscript,⁵ now published, was received. This work describes an investigation employing such a piston pressure device. The results, insofar as radial pressures are concerned, are at complete variance with the findings of this author. They are discussed more fully below.

Cleveland Meeting, October 1949.
TP 2678 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received April 25, 1949; revision received June 28, 1949.

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¹ References are at the end of the paper.

more serious disadvantage is the disturbance caused by the device itself. It would serve to change the forces it was designed to measure. No matter how small the movement of the gauge, when pressure is applied a discontinuity would exist in the wall surface at that point. Due to the stress concentration caused by the hole, abnormal deflections of the die wall would occur around the gauge. During pressing, powder would be forced into the resulting depression. The depression would then become larger with increasing compacting pressure. Powder, not being a fluid, is capable of supporting shear. The ease with which it would flow into the die wall depression to further move the piston is an indication, not of the radial pressure at that point, but of the state of shear retarding the movement. Thus the "pressure" gauge is really a criterion of flowability, and of the capability of the powder to support shear. For these reasons, it was decided that the electric strain method, herein employed, was more reliable, if more indirect. The gauges and lead wires, mounted on the external die wall do not in any way affect the behavior of the metal powder or the die during pressing.

Theory of the Method

THE EFFECT OF RADIAL PRESSURE ON THE DIE WALL

Effect of a Single Small Band of Hydrostatic Pressure

Consider a die which is a thick-walled cylinder of outer radius R_o and inner radius R_i . If over a small finite length L there is a normal pressure P , a tangential strain distribution at the outer wall results. This is shown schematically in Fig 1. The exact shape of the curve may be predicted by an extension of the theory of a semi-infinite beam on an elastic foundation.⁶ This

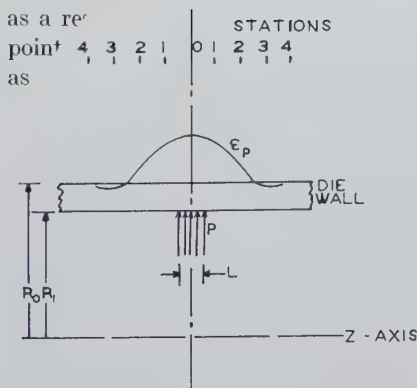


FIG 1—Schematic illustration of strain due to a single increment of pressure.

Note that in all figures, strain abscissa correspond with outer die wall unless otherwise indicated. Z-axis is the axis of the die.

extension has been developed and experimentally verified by MacGregor and Coffin⁷ in their work on symmetrically-loaded thick-walled cylinders. For the calculation of external strains due to increments of pressure the accuracy of their theory is well within 5 pct for very small strains, and within 2 pct for larger strains. Since in our experiments the ends of the tube are not capped, the normal forces in the axial direction are carried by the die plungers, and we thus need not consider the effect of an axial tensile force on the die wall.

Using the above-mentioned theory, values of strain may be calculated along the length of the die at stations a distance L apart (See Fig 1). It is well to note that L should be small enough so that a powder compact of height L will exhibit fairly uniform normal pressure over its length while under compression. The calculations may be verified by filling the die with a hydrostatic fluid in sufficient quantity to occupy a length L in the die, the pressure P being exerted by loading the die plungers in a press. Readings may then be taken on the tangential electric strain gauges.

The calculated tangential strain ϵ_P caused by P is noted at each station.

Values of $F_n = \frac{\epsilon_P}{P}$ may then be calculated, and F_n is called the pressure influence factor. The subscript n indicates the station (distance) from the center of increment L . Since the die is elastic, if the value of P is increased, ϵ_P will increase in proportion. If a measured tangential strain ϵ_{Pn} at station n is caused by an unknown pressure P , then:

$$P = \frac{\epsilon_{Pn}}{F_n} \quad [1]$$

Effect of Several Small Bands of Hydrostatic Pressure

In a compact pressed from both ends, conditions will be symmetrical about the center.⁸ Suppose for the time being that the compact exerts no shear force on the die wall, and that the length of the compact is broken down into five increments, each of length L . Each increment L is assumed to exert uniform radial pressure over its length, as shown in Fig 2. The strain due to each pressure band (increment) is similar in shape to that of a single band. By the principle of elastic superposition the total strain at any point along the die axis is the sum of the five individual strains at that point (see Fig 2). The previously calculated influence factors hold for each of the five individual strain curves. Applying these elastic influence factors to each of the five strain curves, there result five expressions for the total strain due to pressure at stations 0 to 4 inclusive:

$$\begin{aligned} (\epsilon_P)_0 &= F_0 P_0 + 2F_1 P_1 + 2F_2 P_2 \\ (\epsilon_P)_1 &= F_1 P_0 + (F_0 + F_2) P_1 + (F_1 + F_3) P_2 \\ (\epsilon_P)_2 &= F_2 P_0 + (F_1 + F_3) P_1 + (F_0 + F_4) P_2 \\ (\epsilon_P)_3 &= F_3 P_0 + (F_2 + F_4) P_1 + (F_1 + F_5) P_2 \\ (\epsilon_P)_4 &= F_4 P_0 + (F_3 + F_5) P_1 + (F_2 + F_6) P_2 \end{aligned}$$

The above constitute five equations for the three unknowns P_0 , P_1 , and P_2 , any three of which may be used for a solution after measurement of $(\epsilon_P)_0$, $(\epsilon_P)_1$, etc. by the electric strain gauges. A similar equation may be written for the total tangential strain at any point along the die.

EFFECT OF SHEAR ON THE DIE WALL

Effect of a Single Small Band of Uniform Shear

A compact undergoing compression will exert shear force in an axial direction on the inner die wall. Consider a shear stress exerted over an increment of length L in the die. The total resulting force T on the wall is then:

$$T = 2\pi R_i L \tau \quad [3]$$

Fig 3 shows this wall shear force T ; it is statically equivalent to the compressive force B , plus the moment couple $T-C$. From the separate consideration of the couple and the com-



FIG 3—Shear system and equivalent moment-compressive force system.

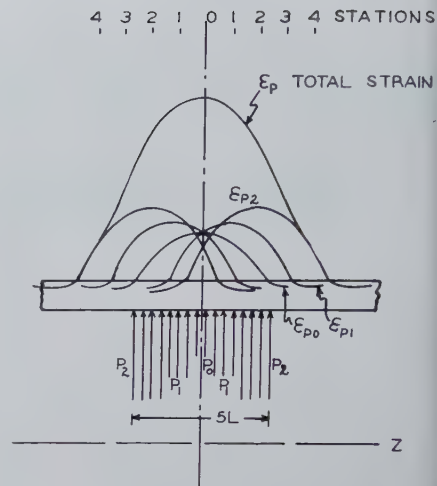


FIG 2—Schematic illustration of strain due to five increments of pressure.

pressive force one may calculate the shape of the strain curve due to the shear stress τ acting over the increment L . The compressive force gives rise to a linearly increasing tangential strain ϵ_r'' throughout the increment L . This strain is then a constant along the remainder of the tube to the end, where an equal and opposite shear force T must be applied to maintain equilibrium. The couple causes the strain ϵ_r' , which is antisymmetrical about the center of L . In effect, to the left of L the tube is constricted. To the right it is bulged. The sum of two curves is ϵ_r , the total tangential strain caused by the shear stress and is shown in Fig 4.

Since the total strain curve due to shear is asymmetrical account must be taken of this in denoting stations. As shown in Fig 4, positive stations will be taken on the side of positive strain; negative stations on the side of negative strain. At each station a shear influence factor $f_n = \frac{\epsilon_r}{\tau}$ is calculated.

On the positive strain side of the shear curve, the strain approaches a constant value (end effect). Correspondingly, for all stations in this region, the shear influence factors will be constant, and will be designated as f_n . The subscript e signifies end effect. Shear influence factors may then be tabulated.

Calculations of shear influence factors may be verified by placing a suitable material, such as metal powder, in the die. It should be of such quantity that a compact of length L will result after compaction to some arbitrary plunger pressure. While maintaining the plunger pressure an axial force T is applied to the circumference at one end of the die (see Fig 5). Such force should not be large enough to cause the

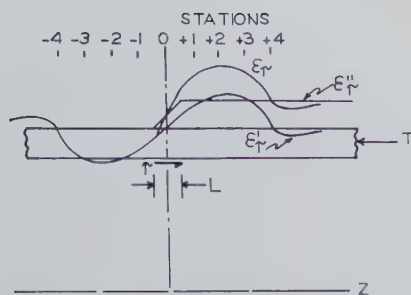


FIG 4—Schematic illustration of strain due to a single increment of shear.

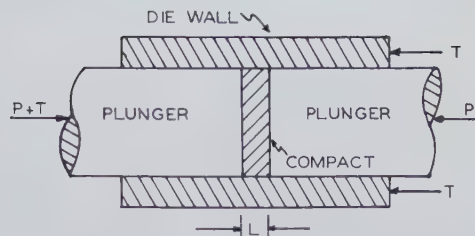


FIG 5—Schematic diagram for the arrangement for a shear check test.

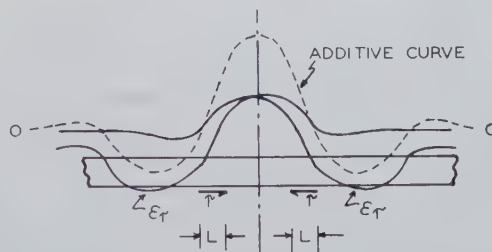


FIG 6—Schematic illustration of the addition of strains due to equal and opposite shear increments. The new strain abscissa is indicated by the line O-O.

die to slide by the compact. Readings may now be taken on the electric strain gauges. Such readings represent the sum of strain due to radial pressure exerted by the compact, and strain due to the applied shear. The shear force T is then removed. Readings of the strain gauges are again taken. Such readings represent strain due to pressure only. Difference between the two sets of readings yields values of strain due to shear. The shear stress is known from Eq 3, and the shear influence factors may then be checked.

Effect of Several Small Uniform Bands of Shear

Here, use is again made of the fact that in a compact pressed from both ends conditions will be symmetrical about the center. According to Kamm et al,⁸ the powder at the center does not move axially during pressing. The wall shear will therefore vary symmetrically about the center, with a value of zero for the center increment.

Consider in such a compact the strains due to shear caused by two shear increments equally and oppositely placed about the center. Strain distributions are schematically illustrated in Fig 6. Unlike the case of the single increment of shear, the die no longer need be supported at one end by a force T in order to be in equilibrium. Instead the compressive (end) effect of one increment is opposed by the compressive effect of the opposite increment. The compressive effect thus exists only between the increments, and does not persist down to the end of the die, as in the single increment case. In effect, the strain abscissa is moved up to 0-0 (see Fig 6), thus sub-

tracting the end effect strain beyond the increments (the portion of the die between either end and the edge of the nearest increment). Mathematically, this is accomplished in subsequent equations by subtracting the influence factor for end effect f_e from all terms involving the addition of strains due to opposite shear increments.

Note that the foregoing correction does not invalidate the principle of superposition. It is necessitated only because the boundary conditions of the force system (with several shear increments now present) are different from the boundary conditions for the single increment of shear. In effect, the force T has been moved from the end of the die to the body of the die.

With this correction in mind, and the fact that the wall shear at the compact center is zero, the expressions may be written (analogous to Eq 2) for total strain due to shear at any point. For five increments these are:

Similar equations may be written for the total strain due to shear at any station.

SOLUTION OF BOUNDARY STRESSES ON THE INTERIOR DIE WALL

In pressing of metal powder compacts from both ends, Eq 2 relates tangential strains with radial pressures on the die wall, and Eq 4 relates strains with shear. When both shears and pressures are present, as is usually the case, the expressions of Eq 1 and 2 are additive for each station.

Terms shown in the boxes are the ones of interest for a compact divided in three increments.

In the above, values of F_n and f_n are calculated. Strain values may be determined by experiment. Solution for the unknown shears and pressures may then be carried out.

Eq 5 treats the powder compact as a

$$\begin{aligned} (\epsilon_r)_0 &= 2(f_{+1} - f_e)\tau_1 + 2(f_{+2} - f_e)\tau_2 \\ (\epsilon_r)_1 &= (f_0 + f_{+2} - f_e)\tau_1 + (f_1 + f_{+3} - f_e)\tau_2 \\ (\epsilon_r)_2 &= (f_{-1} + f_{+3} - f_e)\tau_1 + (f_0 + f_{+4} - f_e)\tau_2 \\ (\epsilon_r)_3 &= (f_{-2} + f_{+4} - f_e)\tau_1 + (f_{-1} + f_{+5} - f_e)\tau_2 \\ (\epsilon_r)_4 &= (f_{-3} + f_{+5} - f_e)\tau_1 + (f_{-2} + f_{+6} - f_e)\tau_2 \end{aligned} \quad [4]$$

$$\begin{aligned} \epsilon_0 &= \boxed{F_0 P_0 + 2F_1 P_1} + 2F_2 P_2 + \boxed{2(f_{+1} - f_e)\tau_1} + 2(f_{+2} - f_e)\tau_2 \\ \epsilon_1 &= \boxed{F_1 P_0 + (F_0 + F_2)P_1} + (F_1 + F_3)P_2 + \boxed{(f_0 + f_{+2} - f_e)\tau_1} + (f_{+1} + f_{+3} - f_e)\tau_2 \\ \epsilon_2 &= \boxed{F_2 P_0 + (F_1 + F_3)P_1} + (F_0 + F_4)P_2 + \boxed{(f_{-1} + f_{+3} - f_e)\tau_1} + (f_0 + f_{+4} - f_e)\tau_2 \\ \epsilon_3 &= \boxed{F_3 P_0 + (F_2 + F_4)P_1} + (F_1 + F_5)P_2 + \boxed{(f_{-2} + f_{+4} - f_e)\tau_1} + (f_{-1} + f_{+5} - f_e)\tau_2 \\ \epsilon_4 &= \boxed{F_4 P_0 + (F_3 + F_5)P_1} + (F_2 + F_6)P_2 + \boxed{(f_{-3} + f_{+5} - f_e)\tau_1} + (f_{-2} + f_{+6} - f_e)\tau_2 \end{aligned} \quad [5]$$

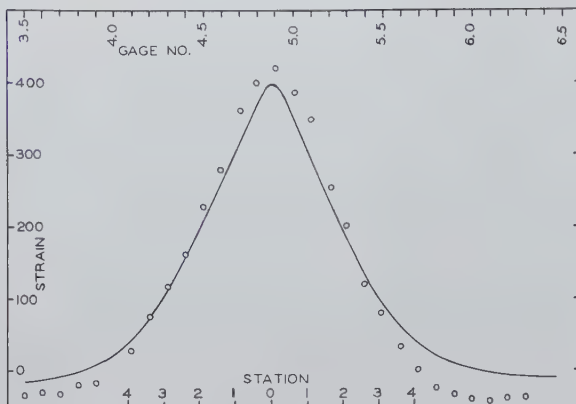


FIG 7—Check tests for 0.20 in. increment of hydrostatic pressure.

Strain in microinches per inch plotted along die length. Solid line indicates theory. Circles indicate experimental values. 55,000 psi pressure.

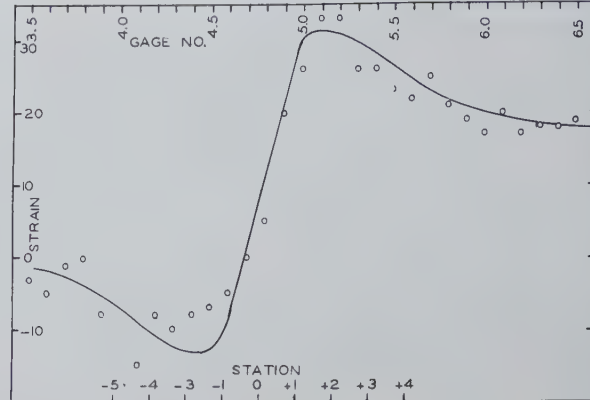


FIG 8—Check test for 0.20 in. increment of shear.

Strain in microinches per inch plotted along die length. Solid line indicates theory. Circles indicate experimental values. 6381 psi shear stress.

series of average pressures and shears, varying in steps, over short increments of length L . For a given length of compact the approximation will be bettered by using a greater number (necessarily an odd number) of increments. A practical consideration limits this number. The labor of solving linear simultaneous equations increases roughly as the square of the number of unknowns. A mathematical consideration also enters. Consider, for instance, the array of numbers making up the coefficients on the right hand side of Eq 5. This array constitutes the determinant of the system of equations. If by chance this determinant has a value near zero, small errors (on the order of 0.01 pct) in the coefficients will cause huge errors (on the order of 100 pct or more) in the answers. Should the determinant approach zero, the answers approach infinity, regardless of other considerations. In either case, the error in the calculated answer may far exceed the magnitude of the true answer.

Equations similar in form to Eq 5 may be written for compacts divided into even numbers of increments. As will be pointed out, such equations were considered and discarded in the course of the present investigations.

Experimental Procedure

For investigation of boundary stresses the die must satisfy two conflicting requirements. It must be strong enough not to fail, yet be built lightly so that it will undergo large elastic strains in use. The die was therefore made ten inches long with external diameter of 2.000 ± 0.0005 in., and internal diameter (diamond lapped) of 1.128 ± 0.0005 in. Two plungers

were used, each 6 in. long and lapped to have 0.001 in. clearance with the die.

The material was an air-hardening, high carbon, high chromium steel (Airdi 150, Crucible Steel Co.), heat-treated to final hardness of Rockwell C 64. The die showed no sign of wear at the conclusion of the investigation.

Tangential strain gauges (Baldwin SR-4, type A-12) were mounted on the outer wall every 0.10 in. along the axial distance of the central portion of the die, and every 0.80 in. near the ends. Alternate gauges were 180° apart on the die. A total of 41 gauges was employed. The die was then placed in a loose-fitting frame, baked in a drying oven, and the gauges then wax-coated to seal out moisture. Two devices were available for strain measurement. The first was a manual SR-4 indicator, and the second an automatic scanning recorder which read and recorded all 41 gauges in less than a minute.

Pressing of compacts was performed on a standard type of testing machine. Lubricated spherical seats were mounted on the top and bottom platens of the press to prevent transmission of bending forces to the die.

After all equipment was assembled, the behavior of the gauges was checked by loading the die internally with oil under hydrostatic pressure. This method was also used to find the modulus of elasticity of the die steel which was $E = 31.65 \times 10^6$ psi. Poisson's ratio was determined as 0.273. Both the constants were necessary for calculation of the influence factors for pressure and shear (see Tables 1 and 2).

Check tests for the influence factors were performed in accordance with the principles previously described. As a hydrostatic medium, paraffin sealed in a thin rubber bag (0.002 in. wall) to

Table 1 . . . Influence Factors for 0.20 In. Increment of Pressure

(L = 0.20 in.)	
Station	F_n
0	7.21614×10^{-3}
1	5.71976
2	3.56168
3	1.83945
4	0.71276

Influence Factors for 0.20 In. Increment of Shear (L = 0.20 in.)

Station	f_n
-4	-1.05417×10^{-3}
-3	-1.60524
-2	-2.00169
-1	-1.75064
0	1.46280
1	4.60424
2	4.85529
3	4.45884
4	3.90777
e	2.85360

Table 2 . . . Influence Factors for 0.33 In. Increment of Pressure

(L = 0.33 in.)	
Station	f_n
0	11.58639×10^{-3}
1	7.12757
2	2.40515
3	0.20805
4	-0.36247

Influence Factors for 0.33 In. Increment of Shear (L = 0.33 in.)

Station	f_n
-4	-0.18367×10^{-3}
-3	-0.96538
-2	-2.37457
-1	-3.37675
0	2.35422
1	8.08519
2	7.08301
3	5.74978
4	4.89211
e	4.70844

prevent leakage was employed. The amount of paraffin was such that it occupied the proper length in the die while under pressure. The center of the paraffin plug was located by measuring the distance which the plungers extended beyond the die. Results of one pressure check test are plotted in Fig 7. For the shear check test, a copper powder compact, exactly 0.20 in. high under 40,000 psi plunger pressure was used. The die was placed in the press, with the compact inside, and with the plungers loaded. A known force was then applied axially to the die wall, in accordance with the previously described principles. (See Fig 5.) Strain readings were then taken, the load re-

oved, and readings taken again. Results for one test appear in Fig 8. The scattering about the theoretical curve in Fig 8 is due largely to the low strain being measured.

In subsequent pressing tests it is certain that no powder was squeezed up into the space between plungers and die wall (except test C-10-a, copper, at 60,000 psi), since the plungers could be pulled out by hand following the tests.

Wall lubrication was used in several of the pressing tests. Stearic acid was one lubricant employed, and was applied by dissolving in benzene, until a supersaturated slurry was formed. Three coats of this slurry were painted on the die wall. Each was allowed to dry thoroughly before application of the next coat. This procedure resulted in a final heavy coating of stearic acid which completely covered the wall. Molybdenum sulphide, when used as a wall lubricant, was applied in similar fashion. It was not, however, possible to build up a heavy coating of this material on the wall. When colloidal graphite (Aquadag) was employed, a very thin coat was painted onto the die wall and dried for two hours by means of an electric fan. A heavy coating of graphite was found to interfere with insertion of the plungers in the die.

Altogether, thirty successful tests were performed. The weight of powder used in each test was adjusted so that the final height of a compact was either 0.20, 0.60, or 1.00 in. From the recorder chart in each case the strain distribution along the length of the die was plotted and stations noted. For a compact with final height of 0.20 in., the average radial pressure was calculated directly by application of the pressure influence factors of Table 1. A compact of 0.60 in. height was broken into three increments. Shear and pressure influence factors of Table 1 were inserted into the boxed-in expressions of Eq 5, resulting in three simultaneous equations. With strain values at stations 0, 1 and 2 (determined from the

test curve) solution for average pressure and shear at each station was carried out.

It had been originally intended to solve the 1.00 in. compacts by the five increment solutions of Eq 5, using values listed in Table 1. However, the value of the determinant of the system was close to zero and the solutions of the equations were therefore meaningless. Equations were then derived for four increments, but in this case the determinant was even closer to zero. Solutions of the 1.00 in. compacts were finally accomplished by treating them as composed of three increments. Method of solution is exactly as described for a 0.60 in. compact, except that influence factors listed in Table 2 are used. Stations, of course, are then 0.33 in. apart, rather than 0.20 in.

Since there is symmetry to the left and right of station 0, strain values for like-numbered stations either side of station 0 should be alike. In the values of strain used in the equations, small differences at such stations were therefore averaged. With such averaging, reproducibility of strain data at any station was within ± 3.6 pct for the smallest values of strain involved. Large values of strain were completely reproducible. This was demonstrated by repeating two pressing experiments. The range of ± 3.6 pct is due to lack of sensitivity in the scanning recorder at low strain readings. The strain gauges themselves are accurate to ± 1 pct. Two other considerations enter into the final accuracy of solutions to the simultaneous expressions of Eq 5. The first is error in the calculated influence factors (Tables 1 and 2). As pointed out, it may amount to ± 5.0 pct for the numerically smaller factors and much less for the larger. The second consideration is the method employed to solve the simultaneous equations.⁹ With the errors in theory and data enumerated, the method¹⁰ employed in the present investigation was found to propagate a maximum possible error

of ± 23 pct in the final calculated values of pressures and shears.

Selected and typical data are presented in Table 3. A total of fourteen pressing tests were performed on type LB (-100 mesh) electrolytic copper powder (U. S. Metals Refining Co.). Eight of these were pressed, without lubrication, to a final height of one inch. Compacting pressures varied from 3900 to 60,000 psi. Of these eight, two (tests C-16-a and C-13-a) were duplicates of other tests. Two compacts, with final heights of 0.20 and 0.60 in., were pressed, without lubrication, at 40,000 psi. Wall lubrication was used in four other compacts, all of 1.00 in. final height. Three of these were pressed at 30,000 psi, and the fourth at 18,600 psi. Stearic acid, molybdenum sulphide, and colloidal graphite (Aquadag) were the lubricants employed.

The second series of three tests was performed on type C (-325 mesh) electrolytic copper powder (U. S. Metals Refining Co.). All of the compacts were pressed to a final height of 1.00 in. without lubrication. Compacting pressures of 7100, 9200 and 18,600 psi were employed.

Carbonyl iron, type L (General Analine and Film Co.) was used for a third series of six compacts. Two each were pressed at 9100, 21,000 and 32,000 psi. Stearic acid was used as a wall lubricant in one of the compacts pressed at each pressure. All final heights were 1.00 in.

For the fourth series, Swedish sponge iron, type MH (-100 mesh Ekstrand and Tholand Co.), was used. Pressures, height, and lubricants were the same as for the six carbonyl iron compacts, so that a direct comparison could be made.

Experimental Results

From data such as are recorded in Table 3, certain conclusions may be drawn immediately. For a constant

Table 3 . . . Tabulation of Typical Data and Results
Copper—Type LB, -100 Mesh

Test No.	Plunger Pressure Psi	Final Height Inches	Wall Lubrication	Strain-microinches per Inch				Weight of Powder Grams	Pressure-Psi		Shear-Psi 1	$\frac{\tau}{P_1}$ Coef. of Friction	Pct of Solid Density	
				0	1		2		P_0	P_1				
					Left	Right	Left	Right						
C-2-a	40000	1.00	None	525	415	400	160	160	118.5	23129	15756	4799	0.304	80.9
C-2-b		Unloading of Test C-2-a		192	150	158	80	86	118.5	9503	7213	-3098	0.430	80.9
C-3-a	40000	0.20	None	135	105	105	60	64	25.6	18711		0	0	87.5
C-4-b	40000	0.60	None	390	336	330	200	214	73.0	32584	12120	4632	0.381	83.2
C-7-c	30000	1.00	None	370	280	286	112	100	105.3	17588	10288	2898	0.282	72.0
C-12-b	30000	1.00	Stearic Acid	377	285	290	133	125	109.2	19722	10837	-887	0.081	74.5
C-14-a	30000	1.00	Molybdenum Sulphide	375	285	270	115	109	107.2	20636	8917	1302	0.146	73.1
C-15-a	30000	1.00	Aquadag	370	275	271	112	104	107.2	20515	8513	1622	0.180	73.1

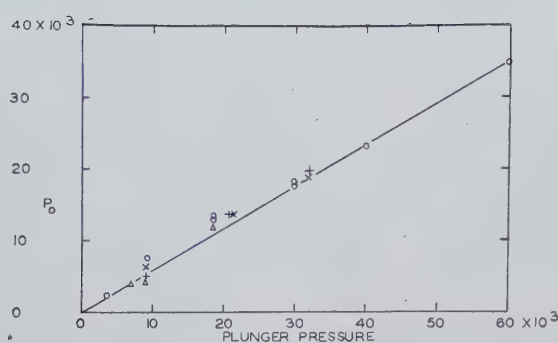


FIG 9—Variation of radial pressure P_0 (center increment) with plunger pressure, both in pounds per square inch.

One inch compacts, no lubrication.
 ○ Copper LB (—100 mesh)
 △ Copper C, (—325 mesh)
 × Swedish Sponge Iron (—100 mesh)
 + Carbonyl Iron L

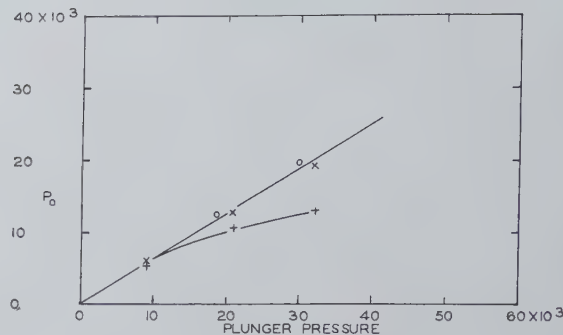


FIG 10—Variation of radial pressure P_0 (center increment) with plunger pressure, both in pounds per square inch.

One inch compacts, stearic acid wall lubrication.
 ○ Copper LB (—100 mesh)
 × Swedish Sponge Iron (—100 mesh)
 + Carbonyl Iron L

final height of compact, average density increases with compacting pressure. Likewise, for constant compacting pressure and die diameter, average density decreases with increasing height of compact. Side wall lubrication is of great importance in securing greater average density of compact. When wall lubrication was employed, the average density of the compact was always greater than that of an unlubricated compact of the same final height and pressed at the same pressure. The foregoing also confirms the work of previous investigators.^{3,8}

In addition, it was found that in all cases but one (Test E-1-3, in which there was evidence of the die acting in bending) the average radial pressure exerted on the die was greater at the center increment of the compact than at the end increments (adjacent to the plungers). In other words, P_0 , the pressure at the center, is greater than P_1 , the pressure at the ends. A possible explanation of this is based on the fact that the most dense part of the compact is at the outer circumference adjacent to each plunger, and the least dense part is at the middle outer circumference.³ Thus the metal powder adjacent to the plungers comes closest to behaving like a solid metal. On the other hand, the central portion of the compact is closer to being an aggregate of metal powder particles. Such an aggregate if placed between two plungers (with no surrounding die wall present) and squeezed, would flow radially, retarded only by the interparticle friction.

A solid plug, if similarly pressed, would offer considerable resistance to any such flow. As a consequence, in a compact undergoing compression from both ends in a die, the central portion, being the least dense, is the most capa-

ble of exerting radial pressure. Of course, the foregoing picture is highly simplified. Strain-hardening of the metal powder particles, change of particle shape, and bonding of the particles during pressing all play a role. The picture is supported, however, by the low radial pressure (18,000 psi for P_0) measured in compact C-3-a (type LB copper powder) which had a final height of 0.20 in. after compaction at 40,000 psi. This compact was pressed to 87.5 pct of solid density. In compact C-2-a, made of the same material and at the same pressure, but with a final height of 1.00 in. the value of the radial pressure P_0 at the central increment was 23,129 psi. Compact C-2-a was only 80.9 pct of final density.

The compact of intermediate length, C-4-b, again of the same material, pressed at the same pressure, but to a final height of 0.60 in., at first examination does not support the above argument. For this compact, the average radial pressure of the center increment is higher than that in either of the two compacts cited above. This is due, it is believed, to compact C-4-b being broken down into 0.20 in. increments for calculation, whereas compact C-2-a was broken into 0.33 in. increments. Since the central portion of a compact has the highest radial pressure, then the shorter the center increment length L over which an average is calculated, the higher such average will be. As a corollary of this statement, it follows that since the end increment has the lowest radial pressure the shorter the length over which it is figured, the lower its average will be. This is the case. The average and radial pressure P_1 for compact C-4-b was 12,120 psi, less than for either of the other two. (In compact C-3-a 0.20 in. high, figured as composed of one increment, P_1 is iden-

tical with P_0 .)

As mentioned previously other investigators,⁵ whose results were received after completion of the present experiments, found a completely different type of radial pressure distribution. Their work was performed on compacts pressed from one end. Their method of measurement employed a piston-type pressure gauge, set in a hole through the die side wall. They state that radial pressure varies from a maximum at the end of a compact adjacent to the moving plunger, to a minimum at the static plunger, in linear fashion. In terms of a compact pressed from both ends, it would mean a V-type distribution of radial pressure, with the point of lowest pressure at the center, and the point of highest pressure next to the plungers. The nature of the results obtained by the above workers may be attributed to the fact that their piston type pressure gauge disturbs the uniform flow of powder and measures the ability of the powder to support shear. Thus as pointed out in the introduction it measures the flow of the powder rather than true radial pressure.

It is unfortunate in the present investigation that the one inch compacts could be treated only as composed of three increments. As a consequence no indication of point to point stress distribution along the length of a compact could be ascertained. Certain general trends are nevertheless evident. Fig 9 shows a plot of average radial pressure P_0 (center increment) versus compacting pressure. It indicates a straight line relationship between radial and compacting pressure for all four metal powders pressed without lubrication.

Fig 10 shows a similar plot, but for tests in which stearic acid wall lubrication was used. Again for copper, type

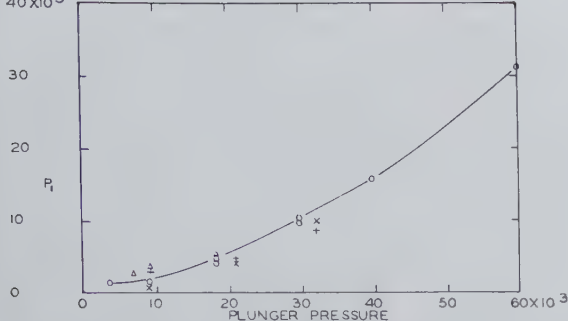


FIG 11—Variation of radial pressure P_1 (end increment) with plunger pressure, both in pounds per square inch.

One inch compacts, no lubrication.
 ○ Copper LB (-100 mesh)
 △ Copper C (-325 mesh)
 × Swedish Sponge Iron (-100 mesh)
 + Carbonyl Iron L

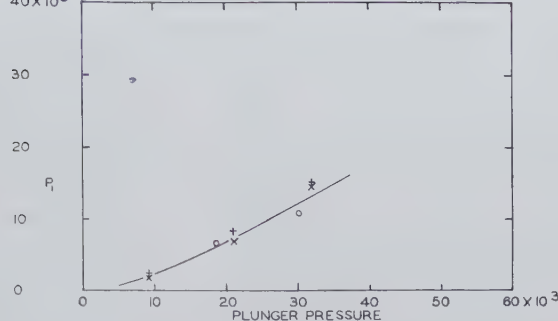


FIG 12—Variation of radial pressure P_1 (end increment) with plunger pressure, both in pounds per square inch.

One inch compacts, stearic acid wall lubrication.
 ○ Copper LB (-100 mesh)
 × Swedish Sponge Iron (-100 mesh)
 + Carbonyl Iron L

C (-100 mesh) and Swedish sponge iron, a straight line relationship exists. The slope of the line is almost identical with that in Fig 9. Within the limits of experimental error, the values of P_0 are equal for like metals (except carbonyl iron), pressed at like pressures in both wall-lubricated and unlubricated compacts. For carbonyl iron, with wall lubrication, the values of P_0 , when plotted as in Fig 10, fall well below the straight line relationship, and well below the P_0 values for unlubricated carbonyl iron powder. If, as has been proposed, radial pressure is in some fashion inversely proportional to densification (as distinguished from density distribution) then one would expect Fig 9 and 10 to have similar plots. It is not clear why carbonyl iron should depart from this. It may be related to the fact that of all the powders here used carbonyl iron compacted with wall lubrication showed the greatest increase in percent of solid density over compaction in the unlubricated state. It thus behaves more like a solid than the other powders used.

Fig 11 and 12 are plots of average radial pressure P_1 (end increment) versus compacting pressure. Fig 11 is for metal powders pressed without lubrication, and Fig 12 for those pressed with stearic acid wall lubrication. Both plots are in the form of shallow curves, slope gradually increasing with plunger pressure. For copper powder, type C, pressed at like pressures, values of P_0 (within the limits of experimental error) are equal for lubricated and unlubricated compacts. For Swedish and carbonyl irons, (especially the latter) pressed with wall lubrication, values of P_0 in Fig 12 appear to be somewhat higher than for the unlubricated state. It cannot be said whether or not the departure is due to error, since it is not a marked deviation. It is possible

that the stearic acid wall lubrication, being in a thick coating, is acting as a semifluid, transmitting pressure to the die wall in a quasihydrostatic fashion.

Some of the most significant results obtained are those concerning average shear stress τ_1 and coefficient of friction on the die wall adjacent to the plungers. These results are for unlubricated pressing to a final height of one inch. (See Fig 13 and 14). For copper, type LB (-100 mesh), the curve of shear stress (Fig 13) rises sharply with steadily increasing slope. The point for 60,000 psi compacting pressure (Test C-10-a) is very low because of extrusion of copper into the space between plunger and die wall. Due to such extrusion a large shear stress was acting on the wall toward the ends of the die (negative shear). This caused the average value of shear stress to be very low. The shear stress curve for copper type C (-325 mesh) follows more or less parallel and below the curve for type LB. There are only three points on this curve, however. The ductility of the copper undoubtedly accounts for the sharp rise of the copper shear curves. At the beginning of pressing, the copper particles have only point contact with the wall. As pressing continues they deform, increasing the area in contact with the die wall, and thus increasing the shear stress acting on that wall. The type C copper, having only fine particles, initially has a smaller total contact area with the die wall and thus a lower shear stress for given plunger pressure. As the fine particles are pressed, they do not pack as well as the type LB copper, which has a wider particle size distribution. Consequently the area of contact with the die wall, while it increases, never reaches (for a given plunger pressure) that of the type LB copper. Thus the curves of shear stress versus plunger

pressure rise sharply, in parallel fashion for the two types of copper powder.

The shear stress plot (Fig 13) for carbonyl iron appears to be a straight line, and that for Swedish iron nearly so. This is because of the lesser ductility of the two metals as compared to copper. While at low compacting pressures the shear stresses for iron are higher than those of copper, this is not true at higher pressures. The wall-particle contact area cannot increase as rapidly. Plots for the two types of iron are nearly parallel, with the carbonyl iron values being higher, even though the Swedish iron is more ductile. This is in part related to the fact that Swedish sponge iron cannot (under like conditions) be compacted to as high an average density as carbonyl iron. The less dense packing of the former contributes to a lower wall-particle contact area. In addition, the wide difference in chemical composition of the two types of irons affects their mechanical behavior.

Fig 14 shows the variation of coefficient of friction with compacting pressure. The plots for all the metal powders (with the exception of copper, type C, for which only three points are known) show an increase, followed by a decrease. The decrease is especially marked for type LB copper due to the low shear stress of test C-10-a before noted. Initial increases, with higher compacting pressure are due to increase in wall-particle contact area by particle deformation. The final decrease is probably caused by the compact reaching a point where strain hardening of the metal slows down the deformation of particles. Thus the wall-particle contact area and shear stress no longer increase rapidly. The coefficient of friction $\frac{\tau_1}{P_1}$ then decreases because the radial pressure P_1 continues to rise

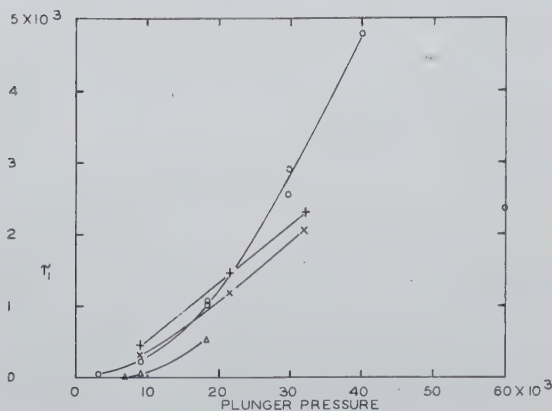


FIG 13—Variation of shear stress τ_1 (end increment) with plunger pressure, both in pounds per square inch.

One inch compacts, no lubrication.
 ○ Copper LB (-100 mesh)
 △ Copper C (-325 mesh)
 × Swedish Sponge Iron (-100 mesh)
 + Carbonyl Iron L

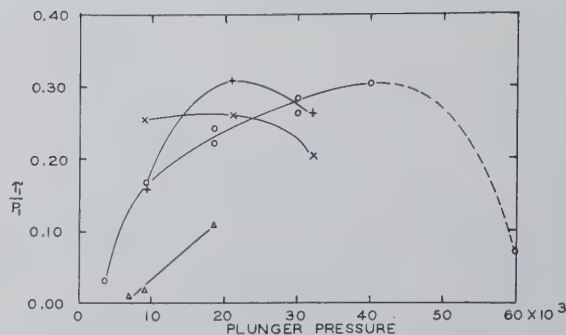


FIG 14—Variation of coefficient of friction $\frac{\tau_1}{P_1}$ (end increment) with plunger pressure, the latter in pounds per square inch.

One inch compacts, no lubrication.
 ○ Copper LB (-100 mesh)
 △ Copper C (-325 mesh)
 × Swedish Sponge Iron (-100 mesh)
 + Carbonyl Iron L

steadily as compacting pressure becomes greater. (See Fig 11 and 12). This explanation is borne out by the curve, in Fig 14, for copper. It strain hardens less rapidly than iron, and therefore the coefficient of friction does not reach a maximum until plunger pressure is quite high. The curves for iron fall off much sooner.

From the curves of shear stress and coefficient of friction versus compacting pressure plotted in Fig 13 and 14, we may draw some conclusions about ejection pressures. For soft ductile metals, the ratio of ejection pressure to compacting pressure should be high. On the other hand, harder, less plastic powders should have a low ratio of ejection pressure to compacting pressure. It is known that tin compacts, for instance, require a greater force for removal from the die; they seem to "drag" on the die wall. Tungsten compacts, pressed under like conditions, are much more easily ejected.

The data for shear and coefficients can be compared to some results obtained by Kamm, Steinberg and Wulff,³ working with lead grids. These authors calculated point-to-point values of the coefficient of friction. One of their compacts, number S-66 (carbonyl iron, type L), was pressed in a die of 0.560 in. diam, at 32,000 psi, from an initial height of 0.55 in. No lubrication was used, and pressing was from one end only. The average coefficient of friction, over the entire final height (obtained by averaging their point-to-point data) was 0.282. The test in the present investigation that compared to their compact was number E-5-d. This was performed on the same material, pressed from both ends at the

same pressure, without lubrication, but to a final height of 1.00 in. The die was 1.128 in. in diam. The average coefficient of friction over the end increment ($L = 0.33$ in.) was 0.261. Even with the entirely different sizes of compact and die and different method of measurement the results are within 8.0 pct of each other. In their point-to-point calculation Kamm et al found the coefficient of friction to be zero adjacent to the static plunger. Therefore in pressing from both ends, the shear stress would be zero at the center of the compact. This was the assumption made in developing the present theory.

The surprising results of the present tests are the relatively small magnitudes of wall shear stresses. The largest is less than 5000 psi. The test results are confirmed, however, by the small magnitude of shear stress that could be supported without slippage between compact and die in the check test, described under *Experimental Procedure*. It was 6381 psi, and its value was ascertained by a direct calibration. Since sliding friction is in general less than static friction, the magnitude of stress encountered in the die must be of that order, or less.

All shear stresses in compacts pressed with stearic acid wall lubrication were in the neighborhood of zero, some in fact being negative. This latter has no physical significance, being due rather to errors of the method. It can only be concluded then that stearic acid, when used as a wall lubricant, reduces wall shear stress to nearly zero. Tests C-12-b, C-14-a, and C-15-a give comparative data for different wall lubricants. All three compacts were type LB copper, pressed at

30,000 psi to a final height of one inch. They show that stearic acid is most effective in reducing shear, while colloidal graphite and molybdenum sulphide are not as good. The last two lubricants are, however, better than none.

A further interesting experiment was performed with compact C-2-a (copper type LB) after the plunger pressure had been released. The compact had been pressed to a final height of one inch, at 40,000 psi. Radial pressure P_0 in the center increment of the compact was 23,129 psi; that in the end increment, P_1 , was 15,756 psi. The average shear τ_1 over the end increment, acting axially on the die wall toward the center of the compact, was 4799 psi. After release of plunger pressure in test C-2-a, another strain record was taken. This showed P_0 had dropped to 9503, P_1 to 7213, and τ_1 to -3098 psi. The negative sign before the shear stress indicates a reversal of direction. The shear stress then acted on the die wall away from the center of the compact. (Or, if looked at the other way around, the shear acting on the compact is toward the center.)

Physically interpreted, the results may explain the cause of lamination cracks in hard powders. When compacting pressure was released, the die (which had been bulged elastically), contracted about the compact. This tended to squeeze it axially from the center of the die towards the ends, thus reversing the direction of shear acting on the die wall. This squeezing action causes the center of the compact to be extruded toward the ends of the die. The circumferential surface of the compact, in contact with the die wall,

cannot move. Thus it is along the axis that such extrusion takes place. The plane of the center of the compact (parallel to the end of the die) is subjected to axial tensile stress. In a hard powder this causes a crack to start, which spreads across the compact resulting in a laminar crack. If such be the case, lamination cracks probably originate while the compact is still in the die, following the release of plunger pressure. They spread to the surface following ejection from the die.

The foregoing explanation of lamination cracks has immediate practical implications. Wall lubrication, by lowering the retarding friction, will permit the circumference of the compact to move with the axial portion, and prevent laminar cracking following the release of plunger pressure. The tendency to crack will thus be reduced. It is well known that hard powders, compacted without lubrication, will almost always form cracks. However, the cracking of compacts made of soft powders, but pressed at extremely high pressures, is also explained. Because of the high pressure, the elastic bulge of the die will be large. After release of plunger pressure, the shear stress acting on the die wall toward the ends of the die is therefore very large. The extruding force, acting to push the axial portion of the compact toward the end of the die, is also large. Consequently, even though the compact is of a comparatively soft, ductile material, a laminar crack will result. The most important implication, however, is of interest to the die designer. Since cracks are caused by the elastic bulge of the die during pressing, the controlling factor in die design is rigidity, not strength. Whereas a die wall ratio (ratio of outer wall diameter to bore diameter) of 3 or 4 might satisfy even the highest requirements for strength, it may very well not result in a die rigid enough to prevent cracking in the compaction of hard powders. With knowledge of radial pressures acting on the interior die wall, it is possible, in the light of existing theory,⁷ to calculate elastic strains and deflections of the die.

Discussion of Results and Conclusions

By analysis of elastic strains of the die during pressing, average shear and radial pressures may be calculated over the length of a compact pressed from

both ends. The results of the analysis aid in forming a picture of the pressing process. During compaction, the powder flows axially through the die, propelled by the plunger. If the die walls are smooth and well-lubricated, the flow will be fairly even across the die and thus a higher average density, and a more even density distribution result. If the die walls are rough, or there is no wall lubrication, frictional effects cause marked retardation of powder movement at the die wall. More flow then takes place in the axial portion than in the circumferential portion. Average density will, as a result, be lower and density distribution will be less even. Die wall friction is therefore one of the most important factors controlling the density distribution of a metal powder compact.

Friction between the compact and the die wall is responsible for shear stresses at the wall of the die. Such stresses are of least magnitude at the middle circumference of the compact (when pressed from both ends) and highest adjacent to the plungers. Radial pressures on the other hand are distributed in a reverse fashion, the maximum stress being at the middle circumference and the minimum at the plungers. The magnitude of the radial pressures are, as to be expected, largely a function of the compacting pressure employed. The one exception found in this work was the extremely fine and hard carbonyl iron powder.

Wall shear stresses during compaction without lubrication are functions of ductility and particle size distribution of the metal powder. Fine particle size (with attendant less dense packing and low wall-particle contact area) in general gives rise to lower shear stresses. Hard metal powders, the particles of which cannot deform to a great degree, will have a relatively low wall-particle contact area for a given compacting pressure. Because of strain hardening, this area does not increase rapidly. For soft, ductile powders, the opposite is true. Consequently, for the latter type, shear stresses rise rapidly as compacting pressure increases.

The same two factors, ductility and particle size distribution, affect the variation with compacting pressure of the coefficient of friction. Radial pressure increases steadily with increasing plunger pressure. Therefore, the coefficient of friction at first increases as wall-particle contact area increases

rapidly and then decreases as strain hardening effects arise. Hard powders usually strain-harden at a faster rate than ductile powders. For the former, therefore, maximum coefficient of friction is reached at a lower compacting pressure.

The results of the present investigation also show that the strength of a die may not necessarily be the controlling factor in the problem of proper die design. A die may adequately withstand, without plastically yielding, the stresses involved in pressing. During pressing, however, the center portion may bulge elastically. Following the release of plunger pressure, the walls try to assume their normal configuration. Die-wall friction then retards movement of the compact surface, yet the center is squeezed in the axial direction. The resulting axial tension starts a laminar crack, which more often spreads to the surface following ejection from the die. While the condition can be partially relieved by wall lubrication, a more satisfactory solution lies in design of the die. The die should be strong enough to not only withstand the stresses, but also to prevent large elastic radial deflections. A thin-walled steel or carbide die is then best mounted in a heavy-walled steel or cast-iron jacket.

The experimental work reported in this paper may be summarized as follows:

1. For a given height of compact, average density increases with compacting pressure. For constant compacting pressure and die diameter, average density decreases with increasing height of compact.

2. Radial pressure on the die wall is greatest at the center of the compact, and least at the ends. Shear stress has the reverse type of distribution.

3. Die-wall friction is the most important single factor controlling density. With no lubrication, frictional shear stresses are a function of particle size distribution and ductility of the metal powder. Wide particle size distribution, with attendant dense packing, causes the shear stresses to be high. Soft metal powders cause shear stresses to increase rapidly with compacting pressure.

4. The coefficient of friction between powder and die wall first increases, then decreases with increasing compacting pressure. Increasing radial pressure, which continues to rise steadily with compacting pressure, is responsible for the decrease in coefficient of friction.

cient of friction. For soft powders (low rate of strain hardening) the coefficient of friction reaches a maximum at a higher compacting pressure than for hard powders.

5. For a die with smooth interior wall, frictional shear stresses may be reduced to practically zero by the use of wall lubricants.

6. Because of elastic deformation of a die during pressing, rigidity rather than strength is a necessary criterion for die design. Elastic bulging of a die results in the formation of laminar cracks.

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Rectangular Hysteresis Loops of Co-Ni-Fe Alloys

R. A. CHEGWIDDEN*

In view of the current interest in magnetic materials having rectangular hysteresis loops, as for example those obtained with the grain oriented 50 nickel 50 iron alloys,† we wish to call attention again to the results obtainable with the permivar (Co-Ni-Fe) alloys heat treated in a magnetic field.

Data taken from direct current measurements made prior to and during World War II give the typical values shown below for two of the permivar alloys. Fig 1 and 2 show the hysteresis loops for these samples.

The first sample above consisted of small rings punched from 0.014 in. sheet stock. These rings were heat treated at 1000°C for one hour in a hydrogen atmosphere and then cooled with the furnace to 750°C at which point they were subjected to a magnetic field of 15 oersteds maintained during cooling to room temperature. The molybdenum permivar sample consisted of a spirally wound core of 0.001 in. tape. The heat treatment was the same except that box annealing methods were used and no hydrogen atmosphere was employed. Even better properties might be expected using hydrogen. The heat treating temperatures and cooling rates are not critical. Rectangular hysteresis loops produced in other permivar alloys by heat treatment in a magnetic field were described earlier.^{1,2}

Properties similar to those described

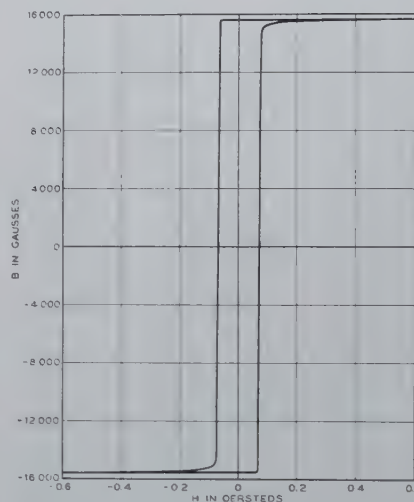


FIG 1—Hysteresis loop for 0.014 in. permivar rings (43 Ni 34 Fe 23 Co) heat treated in a magnetic field.

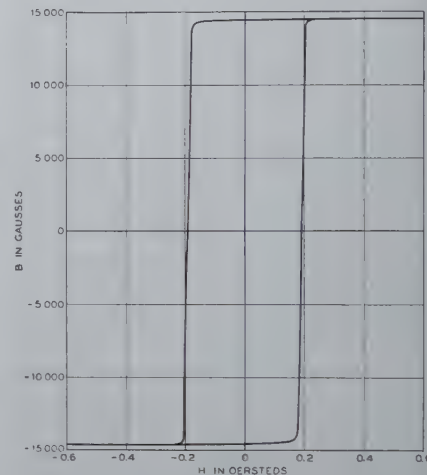


FIG 2—Hysteresis loop for 0.001 in. molybdenum permivar tape core (34 Ni 34 Fe 29 Co 3 Mo) heat treated in a magnetic field.

Nominal Composition	Thickness	B _{max}	B _r	B _r /B _{max}	H _c	Approx. μ _{max}	ρ
43 Ni 34 Fe 23 Co	0.014 in.	15650	15600	0.995	0.07	150,000	20
34 Ni 34 Fe 29 Co 3 Mo	0.001 in.	15200	14500	0.955	0.19	72,000	52

B_{max} = flux density in gauss at H = 20 oersteds

B_r = residual flux density in gauss

H_c = coercive force in oersteds

μ_{max} = maximum permeability

ρ = resistivity in microhm-cm

above are realized in permivars manufactured by conventional methods. Drastic cold working which in practice limits the final thickness and is gen-

erally expensive may thus be avoided. This information may be of interest to those concerned with magnetic alloys and the development of contact rectifiers, magnetic amplifiers and so on.

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* Magnetics Research Dept., Bell Telephone Laboratories.

† Orthonol, Orthonik, Deltamax and similar materials.

¹ References are at the end of the paper.

A Dilatometric Study of the Sintering of Metal Powder Compacts

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Introduction

Dimensional changes generally occur during the sintering of metal powder compacts. These changes may have several causes and their magnitude depends upon the numerous variables involved in the process, such as the nature of the powder, size and shape of the grains, compacting pressure, rate of heating, maximum temperature, length of time at temperature, and furnace atmosphere. Among all the variables, time, temperature, and compacting pressure are probably the most important ones, and the effect of each one of the other variables can be studied separately once the effect of these is well understood.

The most precise method of studying the phenomenon of dimensional changes in a compact consists of heat treating the compacts at various temperatures for different lengths of time. In this method, only very small specimens can be used, in order to achieve rapid rates of heating and cooling. The technique involved in these measurements is very simple, but is time consuming. Another experimental approach to the problem consists of measuring the change in length of a compact as it is being heated at a constant rate. Although the two variables, time and temperature, are combined into a single one (rate of heating), the results obtained by this technique may be of great interest from both practical and theoretical points of view. The purpose of this paper is to describe a systematic investigation of the change in length during sintering of compacts made of one or several metal powders.

Experimental Procedure

The change in length of compacts

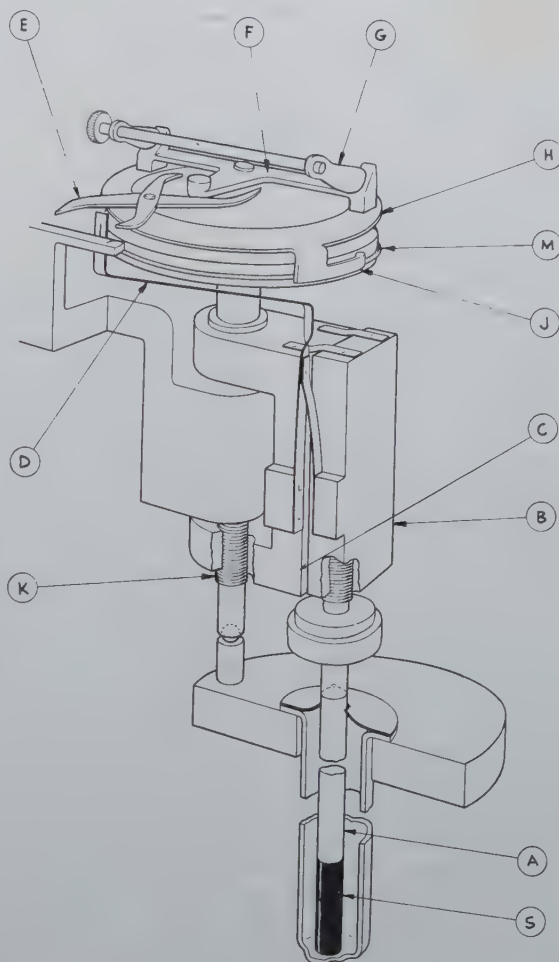


FIG 1—Principle of the dilatometer pick-up mechanism.

during sintering was measured with an automatic recording dilatometer. In this instrument the change in length and the temperature are recorded as

functions of time on two separate charts and the rates of heating and cooling may be varied from 1 to 20°F per min. The temperature may be kept constant at any value up to 2200°F during any predetermined length of time and the change in length recorded as a function of time. Provision is made for atmosphere control inside the fused silica tube containing the specimen. Three different magnifications are provided, so that 1 in. on the chart corresponds to 0.002, 0.005, or 0.010 in. change in length.

The mechanism following the change

Cleveland Meeting, October 1949.
TP 2673 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received May 5, 1949.

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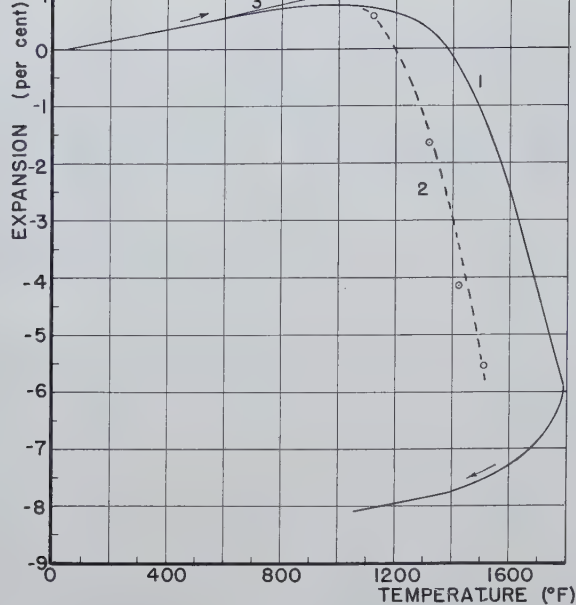


FIG 2—Thermal expansion curve of copper compacted at 20,000 psi.

(1) Rate of heating 7°F per min. (2) Results of isothermal test. (3) Solid copper.

in length of the specimen consists of a mechanical pickup coupled with an electrical transmission to the recorder. The principle of this method is shown in Fig 1. The motion of the specimen is transmitted by the quartz rod to the movable section *B* of a reed gauge mechanism. When the movable section *B* of the gauge is displaced with respect to the fixed section *C*, the wand *D* is displaced from its equilibrium position. The scissors mechanism *E* transfers the movement of this wand as an angular displacement of the clutch bar *F*. The rotating cams *G* reposition the clutch bar *F* to equilibrium position causing rotational movement of the drive disk *H* and the contactor *J*. This motion also rotates the lead screw *K* which causes the fixed section *C* of the reed mechanism to follow the motion previously imposed on the movable section *B* and thus return this mechanism to equilibrium. The position of the contactor *J* on the slide wire *M* is transmitted electrically to the slide wire in the recorder.

The standard specimen used in this investigation was 1 in. long and $\frac{1}{4}$ in. square in cross-section. These specimens were compacted in a hard steel die in which the pressure was applied in a direction perpendicular to the length of the specimen. All tests were performed in an atmosphere of pure hydrogen.

Compacts Made of a Pure Metal

A thermal expansion curve which

may be considered as typical of a pure metal compact is shown in Fig 2. This curve was obtained with a copper specimen compacted at 20,000 psi and made of a classified powder having particle sizes between 74 and 43 microns. The compact was heated at a rate of 7°F per min. The first portion of the curve, up to a temperature of about 600°F represents the normal thermal expansion of copper. As soon as sintering takes place in the compact, shrinkage counteracts the normal thermal expansion and the curve deviates from that of a solid bar of copper. As the temperature increases, shrinkage proceeds faster, the thermal expansion curve reaches a maximum, and finally drops very rapidly.

The change in length occurring during cooling at a constant rate can be measured, and the shape of the expansion curve is also shown in Fig 2. At the beginning of the cooling stage, the contraction of the specimen is larger than that which would correspond to the normal coefficient of thermal expansion of the metal. It is probable that during this period some sintering is still in progress, but at a lesser rate than during the heating period in the same temperature range. As the temperature is lowered the expansion curve follows closely that of a solid metal, showing that all contraction caused by sintering has ceased.

In interpreting the thermal expansion curves of a compact during sintering, it must be remembered that the two variables, time and temperature, are combined into one. Obviously, such curves do not represent equilibrium

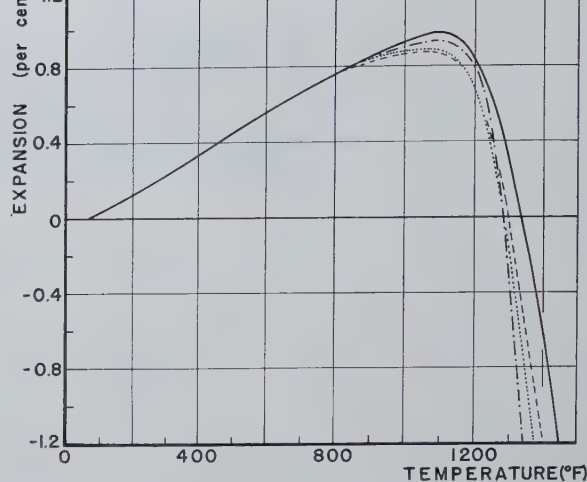


FIG 3—Thermal expansion curves of copper compacted at 80,000 psi.

Recorded at various rates of heating:
— 3°F per min. — 7°F per min. — 11°F per min. — 18°F per min.

conditions, and would be affected by the rate of heating. For rates of heating normally used in practice, however, the effect is relatively small. A series of curves obtained at rates of heating from 3 to 18°F per min is shown in Fig 3. As could be expected, after definite contraction has set in, the expansion curve drops faster for smaller rates of heating. There should be a limit, however, to the shift of the expansion curve toward greater shrinkage as the rate of heating becomes smaller. The limiting curve, which would correspond to an infinitely slow heating can be approximated by making isothermal experiments and measuring the change in length of a compact versus time. Such experiments have been described in a previous paper¹ and the results obtained with a copper powder with particle size ranging from 74 to 43 microns are reproduced in Fig 4, in which the percentage shrinkage is plotted as a function of time. For a given temperature, shrinkage proceeds very fast during the first few hours, and little additional decrease in dimensions is obtained after a long sintering time. From these results it is not possible to decide whether the shrinkage, after the initial period of sintering, tends toward a limiting value or increases at a constant rate. In any case, the shrinkage obtained after a sintering time of the order of 50 hr may be assumed to represent a practical limiting value. Within this approximation, it is possible to trace on the graph of Fig 2 the limiting thermal expansion curve which would correspond to a very small rate of heat-

¹ References are at the end of the paper.

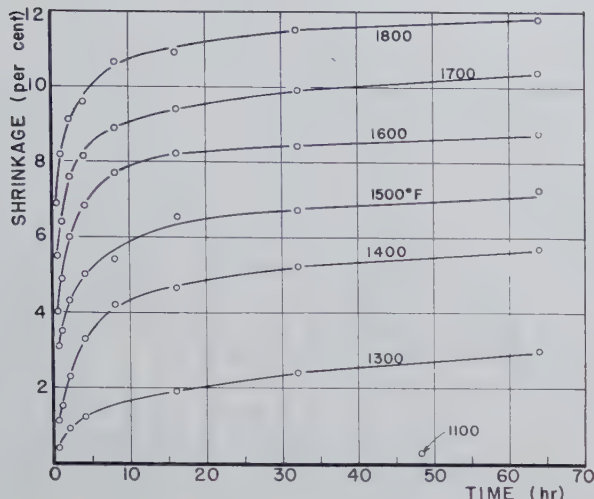


FIG 4—Shrinkage vs. time curves during sintering of copper compacts at various temperature.

ing. Above the temperature at which shrinkage becomes measurable, this curve deviates from that recorded at a rate of 7°F per min but both curves have essentially the same shape. It is particularly interesting to note that both curves separate from the normal thermal expansion curve of solid copper at almost the same temperature. Hence, the thermal expansion curve of a compact recorded at a normal rate of heating can give valuable information on the minimum temperature at which sintering proceeds at a measurable rate.

The influence of the compacting pressure can also be detected by thermal expansion analysis. In general, the expansion curves illustrate clearly the well known fact that the shrinkage of a compact is greater for smaller compacting pressure. As an example, a series of thermal expansion curves recorded for copper, cobalt, and molybdenum compacts prepared at various compacting pressures are shown in Fig 5, 6, and 7, respectively. All the curves are essentially of the type previously described as normal, and for the three metals the effect of increasing the compacting pressure is to increase the temperature at which a given percentage shrinkage is reached.

Most of the pure metal powder compacts studied in this laboratory so far have exhibited the normal type of thermal expansion curves described above. One of the copper powders tested, however, showed an abnormal behavior under compacting pressures above approximately 60,000 psi. At a temperature of about 1600°F, an abnormal expansion or swelling took place. This swelling, almost negligible at 60,000 psi, became greater with

increasing compacting pressure and was about 1.2 pct at 90,000 psi. The thermal expansion curves recorded with this particular copper powder have been published elsewhere.² Thermal expansion curves showing a similar swelling of copper compacts have been obtained by other investigators.³

The first possible explanation for the swelling of a compact is based on the existence of gas pockets enclosed in the compacts by the high compacting pressure. When the temperature is increased, sintering may start before the gas can escape between the particles and the increasing gas pressure may cause plastic flow of the partially sintered metal. In the particular case considered here, the explanation was weakened by the results of experiments made with the same copper powder in which both the pressing of the compact and the recording of the thermal expansion curve were done in vacuum.⁴ The specimens still expanded at high temperature and essentially the same thermal expansion curves were measured. The entrapped gas theory would still remain an explanation, however, if it were supposed that the gas pockets were already inside the particles of powder before pressing and are therefore not affected by vacuum. The existence of such gas inclusions is consistent with the spongy structure of the powder, and the explanation seems at the present time a logical one.

Special Case of Iron Compacts

The dimensional changes taking

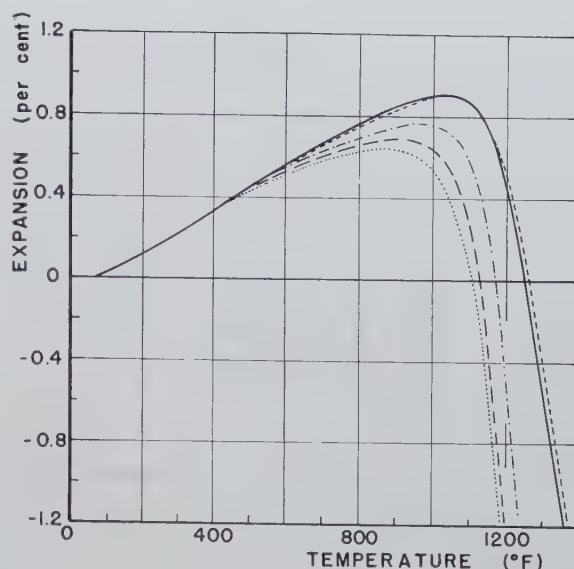


FIG 5—Thermal expansion curves of copper compacted at various pressures and recorded at a rate of heating of 7°F per min.

place during the sintering of iron compacts are greatly influenced by the allotropic change from α to γ iron. The thermal expansion curve (see Fig 8 A) is of the normal type previously described, up to the critical temperature at which an abrupt contraction indicates the transformation from α to γ iron. As soon as this transformation has taken place, the curve starts upward with a slope corresponding approximately to the coefficient of expansion of γ iron. As the temperature increases, shrinkage due to sintering becomes noticeable and the curve deviates from the normal thermal expansion of solid iron, as in the α range.

Before discussing the particular shape of the thermal expansion curve of iron, other features shown in Fig 8 require a short explanation. The influence of compacting pressure, which is quite apparent in the two sets of curves, is in accordance with the previous observation on other pure metal compacts. The influence of the particle size is not very marked, and it would be difficult to base any conclusion from the small difference existing between the two sets of curves, mostly because the two powders are probably different in impurity content. The effect of impurities is clearly shown by the temperature at which the transformation took place. The critical temperature of the fine powder is 1670°F, which is that of pure iron. The coarse powder transformed at about 1570°F, and this low critical temperature is an indication of the presence of impurities, most probably carbon.

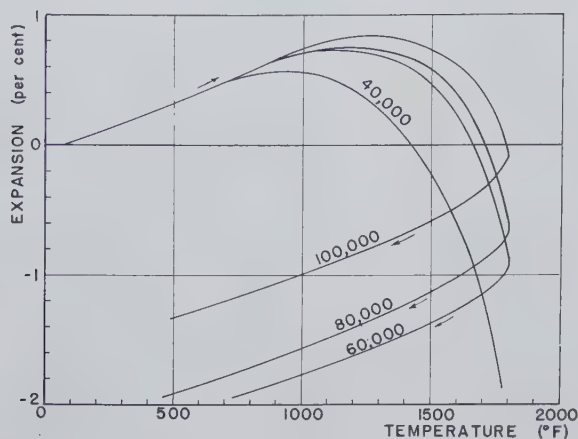


FIG 6—Thermal expansion curves of cobalt compacted at various pressures and recorded at a rate of heating of 7°F per min.

As stated above, the most important feature of the thermal expansion curves of iron compacts is the upward trend of the curve just above the critical temperature. The discontinuity in the rate at which shrinkage proceeds through the critical temperature suggests that near the transformation point the rate of sintering of γ iron is much smaller than that of α iron. In an effort to bring more evidence to this conclusion, the following experiment was performed: An iron compact, prepared with MD-111 powder, was heated in the dilatometer at a rate of 7°F per min. up to a temperature of 1620°F, that is, slightly below the transformation. At this temperature the shrinkage was recorded as a function of time. A second compact was then investigated by the same method, but the isothermal measurements were started at 1690°F, that is, slightly above the $\alpha \rightarrow \gamma$ transformation. The two resulting curves are reproduced in Fig 9, in which the expansion (or shrinkage) is plotted versus time, together with the temperature versus time curves of the two experiments.

Up to 1620°F, the two expansion curves are obviously very close to each other, the slight difference being due to experimental errors and most probably also to the lack of perfect similitude of the two specimens after compacting. During the isothermal part of the test it is quite clear that the sample maintained below the critical temperature contracted at a much faster rate than that maintained above the critical temperature. The net shrinkage one hour after the temperature reached a constant value was 0.85 pct for the specimen heated to 1620°F and only 0.05 pct for the specimen heated to 1690°F. The obvious conclusion is that in the region of the $\alpha \rightarrow \gamma$ transforma-

tion temperature, the rate of sintering is much greater in the α range than in the γ range.

This result may be correlated with observations previously reported by various investigators. For example,⁵ it was shown that both density and tensile strength of sintered iron compacts increase first with sintering temperature, but undergo a marked drop in the vicinity of the critical temperature. Most of the systematic investigations of sintered iron, however, have been made at sintering temperatures above the transformation.⁶ The fact that the rate of shrinkage of a compact heated just below the critical temperature is greater than that above this temperature might be used advantageously in practical application where maximum density and hence maximum tensile strength are desired. In this case, the sintering should be carried on at approximately 1600°F for as long a time as practical, then the temperature should be increased to a higher value, for example 2000°F as was used in Ref. 6. By this technique, advantage could be taken of the rapid rate of shrinkage occurring in the α range, while the final high temperature treatment would achieve a satisfactory sintering.

Case of Alloyed Powders

Apriori, it could be stated that the mechanism of sintering of a compact made of alloyed powder is not essentially different from that of a pure metal compact. As far as the dimensional changes are concerned, this statement has been verified in the cases of a copper-nickel powder (30 pct copper and 70 pct nickel) and a stainless steel powder of the 18-8 type. A

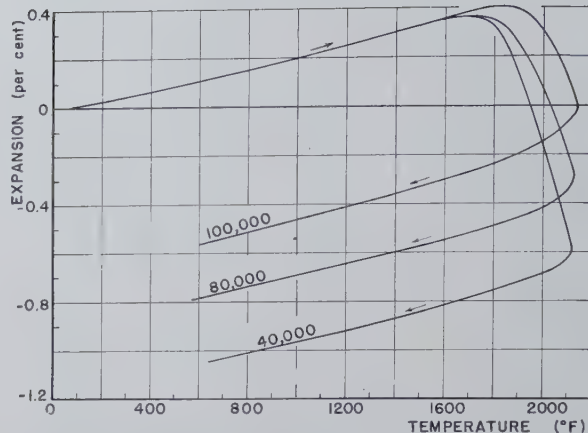


FIG 7—Thermal expansion curves of molybdenum compacted at various pressures and recorded at a rate of heating of 7°F per min.

set of thermal expansion curves of compacts made with the copper-nickel powder is shown in Fig 10, and similar curves for stainless steel powder have previously been published.⁷ All these curves are of the normal type generally encountered with pure metals.

Compacts Containing Two Metal Powders

During sintering of a compact containing two metal powders, relatively large dimensional changes are frequently observed. The swelling of copper-tin and copper-zinc compacts are familiar examples. These dimensional changes may be studied by dilatometric measurements. The interpretation of thermal expansion curves, however, is often more complicated than in the case of a pure metal. To make this interpretation as clear as possible, it is recommended procedure to prepare the mixtures with powders which, tested individually, would have the normal type of thermal expansion curve described in the previous section. If this precaution is taken, any abnormal swelling observed during the sintering of the mixture is most probably due to interaction between the two powders. As a result of numerous experiments performed at this laboratory on the sintering of compacts made of two powders, it was found that the thermal expansion curves during sintering were of two basic types. These two types will first be described and tentative explanations for the dimensional changes will then be discussed.

COPPER-NICKEL COMPACTS

During the sintering of copper-nickel compacts, no abnormal expan-

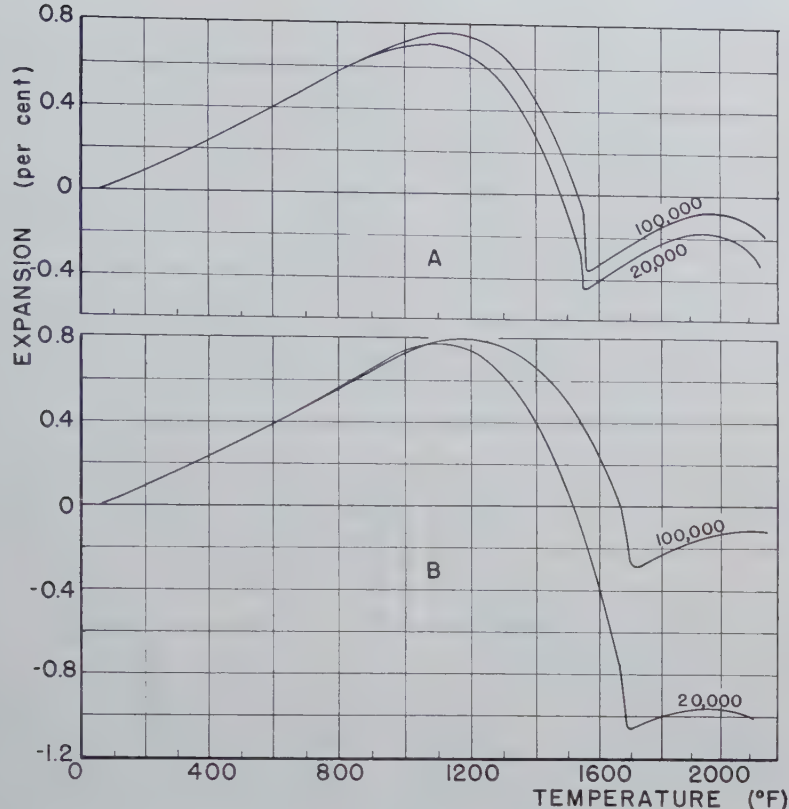


FIG 8—Thermal expansion curves of iron powder compacts recorded at a rate of heating of 7°F per min.
A. Particle sizes between 147 and 104 microns.
B. Particle sizes smaller than 43 microns.

sion has been observed. The thermal expansion curves recorded with powders of various kinds and pressed at compacting pressures varying from 20,000 to 100,000 psi were all of the normal type which is characteristic of each powder taken separately. This study was supplemented by X ray diffraction measurements which showed that simultaneously with the shrinkage of the compact, diffusion takes place and when the compact reaches a temperature of 2200°F at a rate of 6°F per min. its structure is that of the equilibrium solid solution between copper and nickel. The details of this investigation have been published elsewhere.^{2,8}

As far as the present study is concerned, suffice it to say that for copper-nickel compacts the diffusion of the metals into each other does not cause any perturbation in the progression of the normal shrinkage during the process of sintering and the thermal expansion curve is of the type observed for a normal pure powder.

COPPER-ZINC COMPACTS

The salient feature of the thermal expansion curves of copper-zinc compacts during sintering is a large swelling of the compact in a range of temperature from about 300 to 700°F. This has been previously discussed for compacts

containing 70 pct copper and 30 pct zinc.² It was shown that the critical temperature marking the beginning and the end of this abnormal expansion depended upon the fineness of the powders but not upon the compacting pressure. The effect of composition has been recently investigated, and typical thermal expansion curves for compacts containing from 5 to 45 pct zinc are shown in Fig 11. The copper powder

(MD-151) from Metals Disintegrating Co., Elizabeth, N. J.) used for these measurements was 50 pct between 200 and 325 mesh and 50 pct finer than 325 mesh and the zinc (MD-201 from the same source) was all finer than 325 mesh. The curves of Fig 11 indicate clearly that by increasing the percentage of zinc in copper, the magnitude of the swelling of the compact is steadily increased, but the two temperatures between which this swelling occurs do not seem to be affected.

A study of the various phases present during the sintering of 70-30 copper-zinc compacts has been previously made by X ray diffraction.² It was shown that the temperature at which the thermal expansion curve has a sudden increase coincides with a rapid diffusion of zinc into copper and at 300°F the presence of the ϵ phase was already noticed. At all temperatures along the ascending portion of the expansion curve, pure zinc and intermediate phases were present and at the end of the ascending portion (about 550°F) all the zinc had been absorbed into β and α phases. It is therefore apparent that the abnormal thermal expansion between 300 and 550°F coincides with the progressive disappearance of the particles of zinc and their absorption by the particles of copper. A tentative explanation of the increase in volume during the sintering of copper-zinc compacts may be based on the assumption that during the process of diffusion, the zinc particles are absorbed by the copper particles, which, as a result, grow in size leaving cavities in place of the zinc particles. The mass transfer of zinc into copper

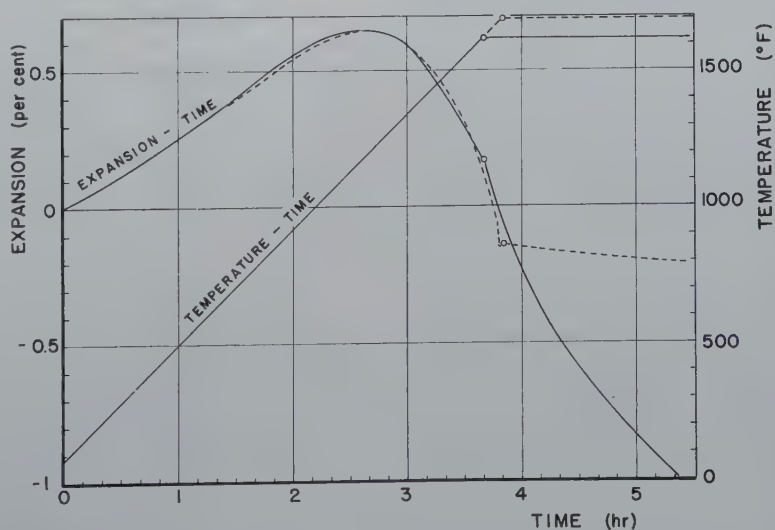


FIG 9—Expansion and temperature vs. time curves for two experiments made with iron powder compacts pressed at 80,000 psi.
— isothermal test in α range
--- isothermal test in γ range

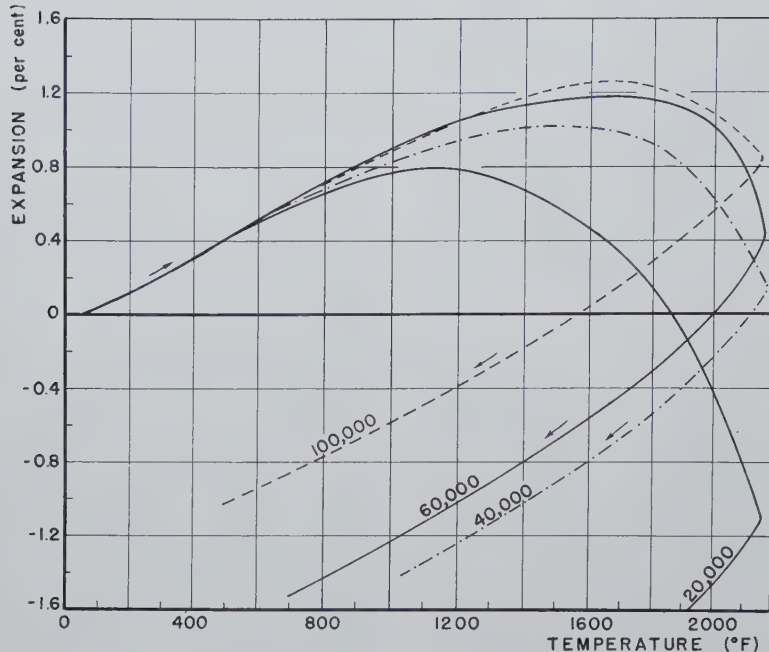


FIG 10—Thermal expansion curves of alloyed copper-nickel powder compacts.

during diffusion is the main hypothesis in this explanation. It may be justified on the basis that zinc is soluble in copper, whereas copper is practically insoluble in zinc. The results of the measurements described above provide a new argument in favor of the absorption of zinc hypothesis. It is indeed quite clear that if zinc moves into the copper particles, cavities are formed and the amount of swelling of the compact should increase steadily with the percentage of zinc. This is exactly what has been found experimentally, as shown by the curves of Fig 11.

FACTORS INFLUENCING THE DIMENSIONAL CHANGES OF COMPACTS CONTAINING TWO POWDERS

The nature of the phase diagram between two metals is a determining factor controlling the occurrence of the swelling of the compact. In the case of copper-nickel, in which no abnormal expansion is observed, the two metals form a complete series of solid solutions, so that either one of the two metals is able to dissolve the other. This condition may be necessary to prevent any swelling, but does not seem to be sufficient. Experiments performed on chromium-molybdenum compacts ranging in composition from 10 to 90 pct, by steps of 10 pct, exhibited a maximum increase in dimensions of 16 pct, as shown in the curve of Fig 12, and yet these two metals form a complete series of solid solutions.⁹

There is therefore another condition

which must be fulfilled to prevent swelling of the compact during diffusion, and it is quite logical to believe that this condition is that the rates of diffusion of either one of the two metals into the other should be approximately the same. This condition is satisfied in the copper-nickel system. Unfortunately it is not possible to apply it at present to the chromium-molybdenum system because the diffusion constants for this pair of metals have not been determined.

The explanations suggested for the swelling of the copper-zinc compacts are supported by several experimental results obtained in this laboratory. The system copper-10 pct tin exhibits a swelling which, by means of X ray diffraction, has also been related to the absorption of tin into copper.² Compacts made of 25 pct molybdenum and 75 pct nickel also have an abnormal thermal expansion. The mechanism is, in this case, similar to that described for the copper-zinc system, since molybdenum is soluble in nickel up to about 35 pct at 2500°F while molybdenum does not dissolve any appreciable amount of nickel and in addition, three intermediate phases are present in the phase diagram.

Conclusions

The results of experiments described in this paper have demonstrated the usefulness of the dilatometric method in studying the sintering of metal powder compacts. In the case of a compact made of a pure metal, the thermal expansion curves recorded on a dilatometer at a constant rate of heating give valuable information on the rate at which shrinkage proceeds as the temperature is increased. In addition, a dilatometric test offers a simple means of detecting the abnormal behavior of some metal powder compacts which exhibit a swelling during sintering.

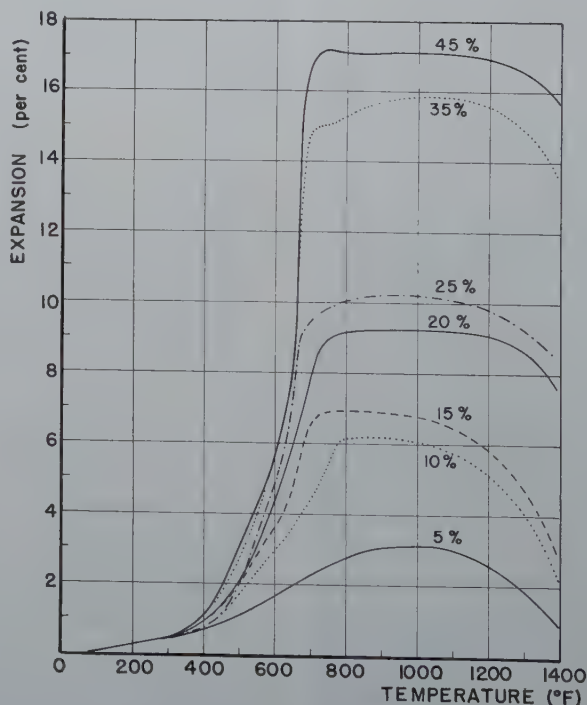


FIG 11—Thermal expansion curves of copper-zinc compacts containing various amounts of zinc and pressed at 40,000 psi.

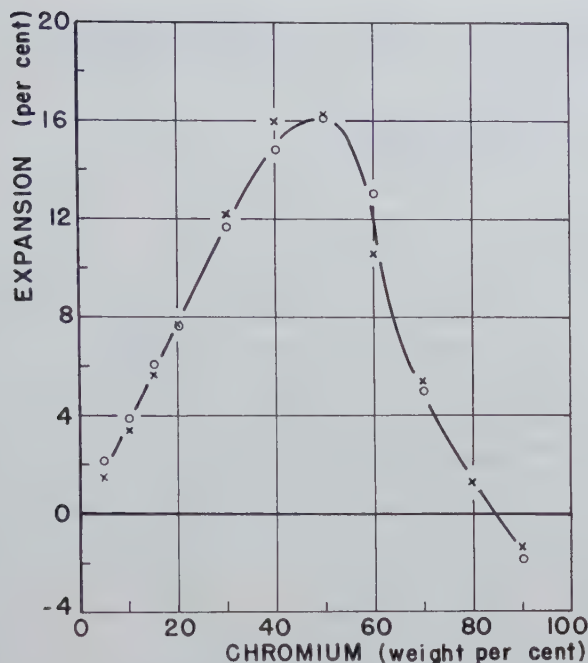


FIG 12—Expansion of chromium-molybdenum compacts measured after sintering for 4 hr at 2500°F.

When a binary alloy is prepared by powder metallurgy by mixing the two powders before pressing, abnormal dimensional changes are likely to take place as the result of diffusion. In most cases, a swelling is observed in the range of temperature in which one of the constituents is absorbed by the other. This critical range of temperature is clearly detected on a thermal expansion curve. Since in the sintering of large compacts the temperature may not be uniform during sintering, the existence of large dimensional changes may result in an objectionable distortion of the compact. There is therefore an advantage in reducing the rate of heating in the critical range of temperatures determined from dilatometric measurements.

As stated before, the thermal expansion curves of a compact during sintering do not provide the kind of data that would contribute most to the search for a theory of sintering, the reason being that the two independent variables, time and temperature, are combined into one, namely the rate of heating. The importance of the dilatometric method should not be mini-

mized, however, in view of the fact that it has already furnished very valuable results, especially in the determination of the critical range of temperatures of compacts containing two metal powders. Considered as a first step, the method is of great help in determining the conditions under which the more reliable, but also more tedious, isothermal experiments should be conducted to bring out the maximum amount of information in the minimum time.

Acknowledgment

This work was done under contract with the Army Ordnance Department, Washington, D. C. The authors wish to thank this agency for the permission to publish the results of this investigation.

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The Isolation of Carbides from High Speed Steel*

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Introduction

Quantitative observations concerning the carbide phases in high speed steel are of importance for two general reasons: (1) the carbides, being inevitable constituents of the final structure, exert a direct influence on the properties of the steel; and (2) a substantial proportion of the total alloy content is tied-up in the carbides, and hence the extent of their solution on austenitizing governs the composition of the steel matrix. The latter relationship has a vital bearing on the response of the steel to tempering as well as on its performance in subsequent service. Accordingly, in the course of a long-term study of the behavior of high speed steels, the authors were confronted with the problem of securing quantitative data on the carbide phases.

The obvious method for acquiring such information is to isolate the carbides from the steel and subject them to chemical, X ray diffraction and other measurements. There are well-known extraction techniques which involve the chemical or electrolytic solution of the less noble matrix (ferrite, martensite or austenite), thus leaving a residue of the carbide phases. However, the results obtained must be scrutinized carefully^{1,2} since the carbides may be affected by the chemical or electrolytic action. It is the purpose of the present paper to describe the experiments leading to an electrolytic-extraction technique for quantitatively isolating the carbides from both annealed and hardened high speed steel. Particular attention is paid to the amount, as well as the composition, of the carbides so that the matrix analysis becomes ascertainable by subtraction from the overall steel composition. Illustrative results are given for the M-2 grade of tungsten-molybdenum steel.

Review of the Literature

The chemical method of dissolving the matrix selectively with respect to the carbides makes use of dilute non-

oxidizing reagents such as hydrochloric or sulphuric acid. Although this simple procedure has led to the determination of the cementite composition,^{3,4} it achieved only limited success because of the interaction between the acid and the carbide residue. Some of the carbides may not only be destroyed in this way, but the hydrogen released is likely to remove part of their carbon as hydrocarbon gases.

The electrolytic technique of isolating carbides has the advantage of rapidly dissolving the specimen (anode) in the presence of less reactive solutions than are practicable with the chemical method. This reduces the possibility of chemical attack on the carbides, and furthermore, the hydrogen evolved during the electrolysis is released at the cathode which is not in close proximity to the carbides. The common electrolytes adopted for this purpose are hydrochloric and sulphuric acids.⁵⁻¹¹ Aqueous solutions of ferrous salts have also been used.^{12,13}

A considerable advance in experimental technique was introduced by Treje and Benedicks¹⁴ who developed a double-compartment cell for electrolytic extraction, the anode and cathode chambers being separated by a porous diaphragm. A solution of 15 pct sodium citrate, 2 pct potassium bromide and 1 pct potassium iodide was selected for the anolyte, while the catholyte consisted of a 10 pct solution

of copper sulphate, with copper serving as the cathode. This type of cell has a number of desirable characteristics:

1. The anolyte has a pH value close to 7, at least at the beginning of the run.
2. The iron that dissolves from the anode-specimen forms a water-soluble complex ion with the citrate, thereby preventing the precipitation of iron hydroxide (which would contaminate the carbide residue) despite the neutrality of the solution.
3. Copper deposition instead of hydrogen evolution occurs at the cathode, and this avoids an increasing concentration of hydroxyl ions which (in an otherwise neutral solution) might cause the precipitation of insoluble hydroxides.
4. Contamination of the anode chamber by copper sulphate is inhibited by the porous diaphragm.

Houdremont and coworkers¹⁵ applied the above method (with the further refinement of excluding oxygen during the electrolysis, washing and drying) to the extraction of carbides from a series of plain carbon steels after various heat treatments. They had quantitative success only with specimens in the annealed condition, and concluded that the size and shape of the carbide particles play an important role in the isolation process, with large spheroids exhibiting the least tendency to decompose during the electrolysis.

Up to the present time, the citrate double-cell has not been used to any extent for isolating the carbides of high alloy steels, apparently on the grounds that the complex carbides are more resistant than cementite to attack in the simpler acid electrolytes. In particular, Bain and Grossmann⁷ and Gulyaev^{10,10a} have employed the hydrochloric acid cell for their investigations of the carbides in high speed steel.* It will be demonstrated here that this type of cell is capable of yielding quantitative results in the case of high speed steel, and actually has certain advantages over the more complicated double cell. However, in order to provide a rigorous test of the quantitative nature of electrolytic procedures for the problem at hand, both methods were studied in considerable detail.

Cleveland Meeting, October 1949.
TP 2625 E. Discussion of this paper
(2 copies) may be sent to *Transactions*
AIME before December 1, 1949.
Manuscript received February 23,
1949.

* This paper is based on a portion
of a thesis submitted by D. J. Blick-
wede in partial fulfillment of the re-
quirements for the degree of Doctor
of Science at the Massachusetts Insti-
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1 References are at the end of the
paper.

Table 1 . . . Analyses of Steels

Steel Designation	C	W	Mo	Cr	V	Si	Mn	S	P
A	0.83	6.09	5.06	4.07	1.85	0.16	0.33	0.008	0.013
B	0.82	5.73	4.53	4.23	1.51	0.26	0.26	0.014	0.019
C	1.09			1.52		0.29	0.32	0.018	0.005
D	1.08			2.83		0.29	0.27	0.010	0.006

Composition of Steels

Four steels, of the compositions given in Table 1, were used for the experiments to be described. Steel A is the present-day grade of M-2 high speed steel. Steel B is the war-time equivalent of the same grade, with reduced alloying elements. Steels C and D are low-alloy laboratory heats being studied for other purposes at M. I. T.

These materials were received in the form of annealed (spheroidized) centerless-ground bars, $\frac{1}{4}$ in. in diam.

The Citrate Cell

PROCEDURE

A number of double electrolytic cells based on that of Treje and Benedicks¹⁴ were built in an attempt to devise a method which would permit rapid, yet quantitative isolations of the carbides from high speed steel. The apparatus found most satisfactory is illustrated in Fig 1. Its principal features are:

1. The anolyte is an aqueous solution of 15 pct sodium citrate, 1 pct potassium bromide and 0.1 pct potassium iodide, having a pH of 7.0 ± 0.2 . The catholyte is an aqueous solution of 10 pct copper sulphate.

2. The specimens to be dissolved are 5 in. long by $\frac{1}{4}$ in. diam and are supported in pairs from the bottom on the fixture shown in Fig 2. These rods form the anode of the cell, and are rotated at 1 rpm during electrolysis to promote even solution.

3. The anode and cathode compartments are separated by a porous cylinder made by cutting the bottom off a porcelain cup used for Daniel cells.

4. Continuous removal of the iron-rich anolyte and replenishment with fresh solution is necessary to prevent the anolyte from becoming too acidic during the course of electrolysis. The cell in Fig 1 provides for such replenishment automatically. With this arrangement, the pH of the anolyte drops only to about 6.6 after a 24 amp-hr run. Without it, the pH decreases to 3.6 over the same period.

After a normal run of 24 hr at 1 amp,* the two specimens are not completely dissolved, and in the case of the annealed steels, a large proportion of the carbides adheres to the remainder of the specimens. The specimens are removed from the cell, and the attached carbides are scraped into a beaker with a rubber "policeman" and a jet of water. The syphon that

supplies fresh anolyte from the reservoir is shut off, and the solution in the anode chamber is drawn through the alundum filter crucible (Fig 1) with the aid of an aspirator pump. When the anolyte level drops below the ground-glass joint just above the filter crucible, the joint is broken and the filter assembly is lowered away from the apparatus. The carbides in the aforesaid beaker are transferred to the filter crucible, and the entire residue thus collected is washed with 300 cc of distilled water, followed by 50 cc of 1:20 ammonium hydroxide* and finally 500 cc of distilled water. The filter crucible and its contents are then dried in vacuum at 165°F (60°C) for 12 to 14 hr.

The carbide content of the steel is taken as:

$$\text{Wt. pct of carbides} = \frac{(\text{Wt. of filter crucible} + \text{contents}) - (\text{Wt. of filter crucible})}{(\text{Wt. of specimens before run}) - (\text{Wt. of specimens after run})} \times 100 \quad [1]$$

* The ammonium hydroxide wash is used to dissolve any tungsten acid which may possibly form from the tungsten originally in the steel matrix.

* Current density = 0.14 amp per sq in.

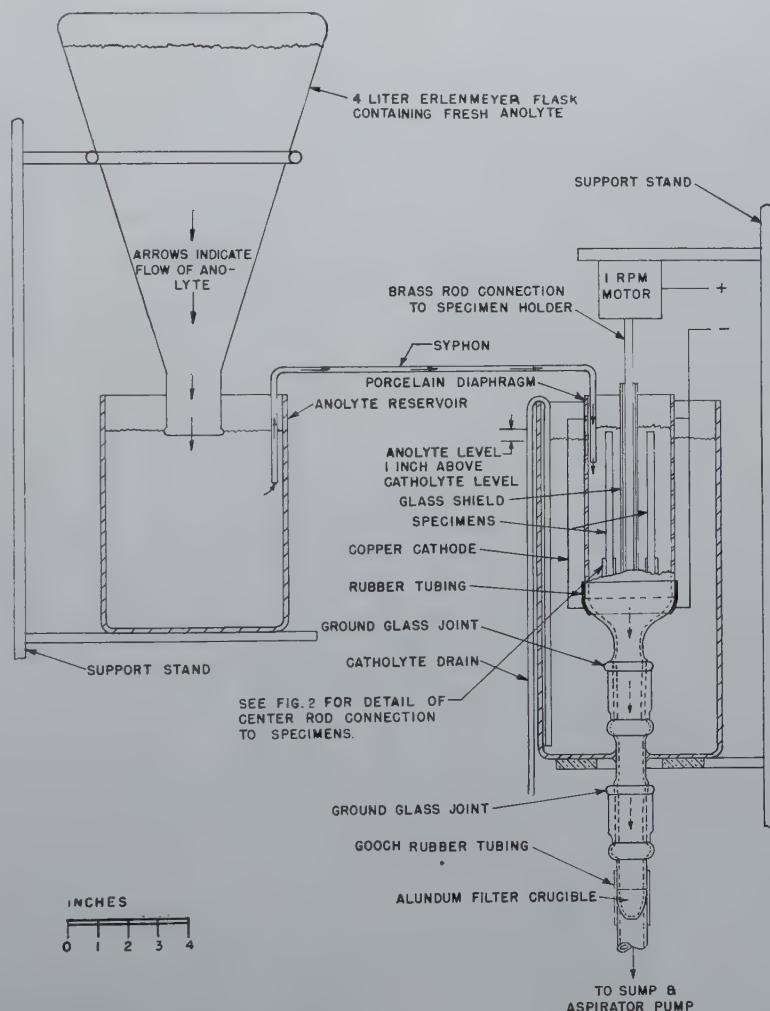


FIG 1—Schematic diagram of sodium citrate cell for electrolytic isolation of carbides. Anolyte: aqueous solution of 15 pct sodium citrate, 1 pct potassium bromide and 0.1 pct potassium iodide. Catholyte: aqueous solution of 10 pct copper sulphate.

The amounts and compositions of the residues obtained by citrate-cell extractions of annealed steels B, C and D in the annealed condition are listed in Table 2.

The fact that the carbon content of the residues isolated from the low alloy steels C and D closely approximates the theoretical composition of cementite (6.67 wt. pct) offers one criterion that the cell is quantitative, even for low alloy carbides. In addition, it is known that virtually all the carbon in the three annealed steels is tied up in the carbides. This circumstance may be employed as a second criterion for the quantitative-ness of the isolation, since the amount of carbon in the residue can be compared with that originally existing in the steel as electrolyzed:

Fraction of carbon of steel found in residue

$$= \frac{(\text{Wt. of residue}) \times (\text{Wt. pct carbon in residue})}{(\text{Wt. of steel dissolved}) \times (\text{Wt. pct carbon in steel})} = \frac{(\text{Wt. pct residue})}{100} \times \frac{(\text{Wt. pct carbon in residue})}{(\text{Wt. pct carbon in steel})} \quad [2]$$

On this basis, the data of Table 2 demonstrate that all of the carbon in each steel is accounted for in the extracted residue.

Steel A did not dissolve like the other three during electrolysis. Rather, a porous skin of partially attacked metal remained around the specimens after the runs. Inasmuch as it was difficult to wash the adhering carbides from beneath this skin, the results ob-

Table 2 . . . Amounts and Compositions of Residues Obtained from Isolations of Annealed Steels in Sodium Citrate Cell*

Steel Designation	Weight Pct Residue	Composition of Residue—Weight Pct						Fraction of Carbon of Steel Found in Residue
		C	W	Mo	Cr	V	Fe	
B	21.0	3.84	25.76	18.98	11.44	6.56	27.75	0.98
C	17.0	6.49			8.36		83.70	1.01
D	16.3	6.65			15.58		77.00	1.00

* These investigations did not include M-2 high speed steel.

tained for steel A are not considered sufficiently accurate to be included in Table 2. Numerous attempts were made to eliminate the undissolved skin by carefully degreasing the specimens, altering the surface finish, and varying the current density, but no success was achieved. The method devised for coping with this troublesome occurrence will be discussed later in the paper.

RESULTS ON HARDENED STEELS

To test the behavior of hardened

Table 3 . . . Comparison of Amounts of Residue Obtained in Two Types of Electrolytic Cells from Steel B Quenched from Various Temperatures

Temp. °F	Wt Pct Residue	
	Sodium Citrate Cell	Hydrochloric Acid Cell
Annealed	20.9	21.4
1700	17.2	18.1
1900	16.9*	16.0
2100	18.3*	14.7
2200	21.4*	9.9†

* Peeling occurred, no magnetic separation applied.
† Peeling occurred, but magnetic separation was applied.

high speed steel in the citrate cell, specimens of steel B were austenitized in a salt bath for 10 min. at 1700, 1900, 2100 and 2200°F (925, 1040, 1150 and 1205°C), and quenched in oil at room temperature. They were then cleaned with emery, degreased and electrolyzed. Some of the resulting data are given in Table 3 column 2.

According to these findings, the residue percentage decreases as the

austenitizing temperature is raised to 1900°F (1040°C), and then increases with further increase in temperature. The latter trend is distinctly anomalous, and indicates that the cell operation becomes unreliable when the austenitizing temperature is too high. It was found that somewhat the same phenomenon occurs during the electrolysis of quenched steel B as previously described for annealed steel A; that is, a tenuous layer of metal remains undissolved. However, unlike the case of the annealed steel, the metallic skin tends to split during the run (probably due to the relief of quenching stresses while the electrolysis proceeds), and drops into the filter crucible along with the isolated carbides. This undesirable state of affairs seems to become more pronounced as the alloy content of the steel matrix is increased, and accounts for the erroneous results after austenitizing at the higher temperatures.

The authors are not aware that this "peeling" phenomenon has been observed before in the electrolysis of high speed steel, but Gulyaev¹⁰ has mentioned that it occurs with high-alloy chromium steels, electrolyzed in 1:20 hydrochloric acid. He further states that it may be eliminated by using more concentrated acid. Consequently, experiments were undertaken in the present work with hydrochloric acid cells in an effort to avoid the "peeling" of high speed steel. At the same time,

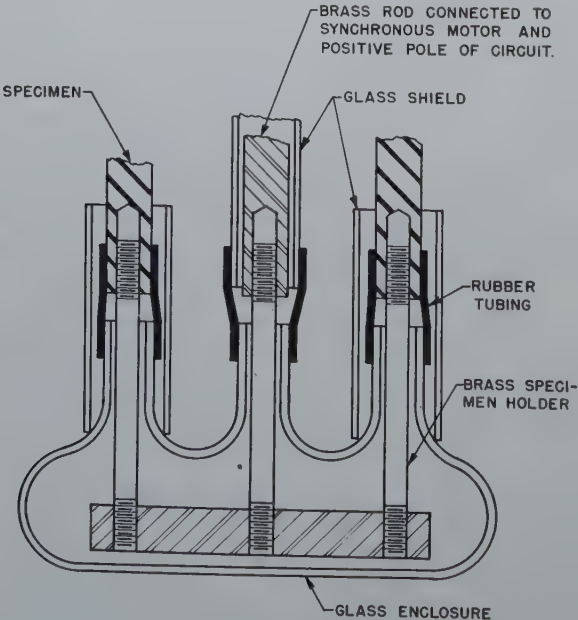


FIG 2—Fixture for holding and rotating specimens in electrolytic cell of Fig 1.

because of the increased danger of carbide attack by the electrolyte in this type of cell, the quantitative aspects of the isolations were cross-checked by lineal analysis¹⁶ and density measurements as well as by chemical analysis.

The Hydrochloric Acid Cell

PROCEDURE

A schematic drawing of the hydrochloric acid cell finally adopted is shown in Fig 3. The anode is a single specimen 5 in. long by 1/4 in. diam, the general arrangement being quite similar to that used by Bain and Grossmann.⁷ Satisfactory runs are carried out at 0.5 amp* for 48 hr in an electrolyte of 1 part hydrochloric acid (sp. gr. = 1.19) and 10 parts distilled water. About half the electrolyte is replaced with fresh solution at the end of 24 hr; otherwise iron may deposit loosely on the cathode and fall into the carbide residue when the cathode is disturbed.

When hardened specimens of steel B are electrolyzed in this simple cell, it is found that the peeling phenomenon occurs only with austenitizing temperatures above 2100°F (1150°C). Thus, although the acid cell does not completely eliminate this interfering condition, it does help appreciably.

The particles of the undissolved skin that contaminate the carbide residue are ferromagnetic and are strongly attracted to a permanent magnet. Fortunately the carbides themselves are nonferromagnetic and this circumstance provides the basis for a method of separation in those runs where the peeling occurs. After electrolysis the spent electrolyte is decanted from the residue in the cell-beaker through a fine-porosity fritted-glass filter crucible, while an Alnico-5 horseshoe magnet is held under the spout of the beaker to prevent magnetic particles from going into the filter. The residue retained in the cell-beaker is then transferred to a smaller beaker together with the particles adhering to the specimen. About 50 cc of 1:20 hydrochloric acid is added to form a slurry, and any large agglomerates of the residue are dispersed with a glass rod. Then, the Alnico-5 magnet enclosed in a thin-walled lucite box is immersed in the slurry, withdrawn and the liquid is allowed to drain back into the beaker. The lucite box is next held over a third beaker, the magnet is removed and the particles adhering to

the box are washed into the third beaker with a stream of distilled water. This operation is repeated several times until no magnetic particles can be withdrawn from the original residue.

Then the same procedure is carried out on the magnetic residue thus transferred, and the nonmagnetic particles finally remaining are added to the first batch of nonmagnetic residue. This is filtered, washed (200 cc of 1:20 hydrochloric acid and 100 cc of distilled water in that order) and dried in vacuum at 165°F (60°C). The magnetic residue, consisting of relatively coarse particles of undissolved steel, settles readily and requires no filtering. The supernatant water is merely decanted off and the residue is washed with alcohol and ether, after which it is dried in vacuum.

The carbide content of the steel is calculated as:

$$\begin{aligned} \text{Wt. pct carbides} = & \frac{(\text{Wt. of filter crucible} + \text{nonmagnetic residue}) - (\text{Wt. of filter crucible})}{(\text{Wt. of specimen before run}) - (\text{Wt. of specimen after run})} \\ & - (\text{Wt. of magnetic residue}) \times 100 \quad [3] \end{aligned}$$

The magnetic separation described above requires due care but is entirely practicable when applied in conjunction with the hydrochloric acid cell. This is not the case with the citrate cell because of the continuous-filtration feature. In other words, the complexity of the citrate cell is hardly worth contending with, if magnetic separation is necessary—provided, of course, that the hydrochloric acid cell can be shown to yield quantitative results.

RESULTS ON ANNEALED AND HARDENED SPECIMENS

Table 3 offers a comparison of extractions performed on steel B with both the citrate and hydrochloric acid cells. These data cast a favorable light on the acid cell, at least for studies on high speed steel, because it not only alleviates the tendency toward peeling but gives good agreement with the citrate cell in those cases where peeling does not interfere. Where peeling does occur, however, it remains to test the quantitiveness of the procedure in which the magnetic process is used to correct for it. Steel A was selected for this purpose.

Weight percentages and compositions of the carbides isolated from steel A are given in Table 4. The heat treated specimens were austenitized for 10 min. in a salt bath at the indicated temperatures and quenched in

Table 4 . . . Weight Percentage and Composition of Carbides Obtained from Steel A in Hydrochloric Acid Cell

Austenitizing Temp. °F	Weight Pct Carbides	Composition of Carbides, Wt Pct					
		C	W	Mo	Cr	V	Fe
Annealed	21.7	3.72	27.3	21.4	9.34	8.05	Bal
1700	19.5	3.90	29.5	22.8	5.28	7.70	Bal
1900	17.3	3.97	31.9	23.7	4.26	7.38	Bal
2000	16.2	3.69	31.6	22.8	2.83	6.73	Bal
2100	14.8*	3.60	30.5	21.5	2.31	7.69	Bal
2150	13.3*	3.60	31.6	22.6	1.72	6.93	Bal
2200	11.6*	3.22	32.3	23.8	2.31	6.02	Bal
2250	10.7*	3.12	22.9	32.1	1.81	5.22	Bal

* Peeling occurred, but magnetic separation was applied.

oil. It is interesting to note that steel A as annealed does not exhibit an undissolved skin when electrolyzed in the hydrochloric acid cell, unlike the results obtained with the citrate cell. Furthermore, virtually all the carbon of the annealed steel is accounted for

in the extracted carbides (using Eq 2: $\frac{21.7}{100} \times \frac{3.72}{0.83} = 0.97$).

It might be possible to find all the carbon in the residue even if the carbides were partially decomposed by the hydrochloric acid because free carbon could be precipitated. Hence, the results were checked independently by density measurements and lineal analysis. It can be shown that

$$\begin{aligned} \text{Density of carbides} &= (\text{Density of steel}) \times \\ & \frac{(\text{Wt. pct of carbides})}{(\text{Vol. pct of carbides})} \quad [4] \end{aligned}$$

In addition to the weight percentage of carbides in the steel as determined by extraction, the other three quantities are directly measurable: the density of the carbides by the pycnometer method,¹⁷ density of the steel by the loss-of-weight in water,¹⁸ and volume percentage of the carbides by lineal analysis¹⁶ on the butt ends of the electrolyzed specimens. Table 5 shows a comparison of the density of the carbides from annealed steel A, as measured and as calculated from Eq 4. The close agreement indicates that, as far as the annealed steel is concerned, the hydrochloric acid cell not only isolates the carbides quantitatively, but leaves them intact.

Unfortunately, in the case of the hardened specimens, the extracted carbides are found to be contaminated

* Current density = 0.28 amp per sq in.

with free carbon which materially reduces the measured density of the carbides. This trend is shown by the

Table 5 . . . Comparison of Measured and Calculated Density of Carbides Obtained from Annealed Steel A in Hydrochloric Acid Cell

Austenitizing Temp. °F	Weight Pct of Carbides*	Volume Pct Carbides	Density of Steel	Density of Carbides Measured	Density of Carbides Calculated
Annealed	21.7	23.2	8.19	7.71	7.65
1900	17.3	16.3	8.08	6.73	8.58
2000	16.2	12.0	8.06	5.54	10.90

* Average of 2-4 runs.

data in Table 5. The true density of the carbides increases with the austenitizing temperature in accordance with the calculated values, but the measured density changes erroneously in the opposite direction. In view of the excellent correlation obtained with the annealed steel, it is believed that the free carbon in the residues of the quenched specimens comes from the martensite-austenite matrix and not from decomposition of the carbides themselves. Attempts to determine the amount of free carbon in the residues by chemical and differential oxidation techniques were unsuccessful. Accordingly, the carbon contents shown in Table 4 for the hardened specimens are undoubtedly somewhat too high and should be regarded as uncertain.*

CORRELATION WITH FILTRATE ANALYSES

Since the foregoing density determinations shed little light on the quantitiveness of the cell operation with hardened specimens, this approach cannot be used for testing the efficacy of the magnetic separation when peeling occurs. Consequently, an analysis was made of the filtrate remaining from one of the extractions in which abundant peeling took place. The specimen (steel A) was austenitized at 2200°F (1205°C), electrolyzed in the hydrochloric acid cell, and the undissolved particles of steel in the residue were separated magnetically. From the quantity of the filtrate and its composition, the weights of tungsten, molybdenum, chromium and vanadium dissolved from the steel

were calculated. Similarly, the weights of these elements in the nonmagnetic residue were also determined.

The resulting weight balance is given in Table 6. It is evident that there is satisfactory agreement between the weight of each element in the steel electrolyzed and the sum of the respective weights in the filtrate and carbides. Thus the magnetic separation process is suitably quantitative. A weight balance based on carbon is not feasible because part of the matrix carbon is lost from the cell in gaseous form and part reports in the residue as free carbon. The fact that the other four elements check each other so well indicates that there is not enough free carbon to affect the weight percentage of the residue materially. The maximum error in the weight percentage of the residue, and therefore in the alloy analyses, due to the free carbon and the magnetic-separation technique appears to be no greater than 1 part in 20.

However, the error in the carbon analyses of the carbides may be 5 or

Table 6 . . . Comparison of Residue and Filtrate Compositions with Composition of Steel A, Quenched from 2200°F (1205°C) and Electrolyzed in 1:10 Hydrochloric Acid (Residue = 12.6 pct by weight)

Element	A Gm Element in Filtrate per 100 Gm Steel Electrolyzed	Composition of Residue Wt. Pct	B Gm Element in Residue per 100 Gm Steel Electrolyzed	A + B	Composition of Steel Wt. Pct
W	1.81	32.3	4.07	5.88	6.09
Mo	2.02	23.8	3.00	5.02	5.06
Cr	3.58	2.31	0.29	3.87	4.07
V	1.05	6.02	0.76	1.81	1.85

10 times larger because of the uncertainty in the amount of free carbon.

Despite the advantages of the hydrochloric acid cell over the more complicated citrate cell for these studies on high speed steel, it should be emphasized that the latter seems to give more reliable results when cementite is present. It was pointed out under THE CITRATE CELL—Results on Annealed Specimens, that the citrate cell operates quantitatively for the cementite in annealed low alloy steels. This is not the case with the hydrochloric acid cell. For example, an annealed steel with 1.11 pct carbon

and 4.11 pct chromium yielded a residue of 8.2 wt. pct of the steel electrolyzed, and the composition was: 8.13 pct carbon, 28.50 pct chromium and 61.38 pct iron. Thus, less than two-thirds of the carbon in the steel could be accounted for in the residue $\left(\frac{8.2}{100} \times \frac{8.13}{1.11} = 0.60\right)$. Evidently the low alloy cementite was partially decomposed during the acid electrolysis. However, since cementite is not found in the M-2 high speed steel under consideration even when the citrate cell is used, the hydrochloric acid cell can be adopted with reasonable assurance for the work at hand.

The Carbides in M-2 High Speed Steel

NATURE OF THE CARBIDES

X ray diffraction analysis of the residues extracted from steels A and B indicates that three carbide phases exist in M-2 high speed steel. These may be designated here as M_6C , $M_{23}C_6$ and MC, the M representing the sum of the metal atoms.

M_6C is a face-centered cubic carbide with a lattice parameter of 11.04₄ A.U. It is identical with the (Fe, W, Mo, Cr, V)₆C phase commonly found in high speed steels.^{19,20}

$M_{23}C_6$ is a face-centered cubic carbide with a lattice parameter of 10.56₄ A.U. Its diffraction pattern also corresponds to that of $Cr_{23}C_6$ as determined by Westgren.^{21,22} Like the M_6C , this phase is capable of dissolving iron, tungsten, vanadium and molybdenum, along with the chromium.

MC is a face-centered cubic carbide with a lattice parameter of 4.18 A.U. It is essentially a vanadium-rich carbide.^{23,24}

The above carbides are present in M-2 high speed steel in the annealed condition and after austenitizing. Therefore, the observed weight percentages and compositions of the extracted residues are not representative of single carbides, but of carbide mixtures. The lattice parameters of these carbides are not appreciably affected by austenitizing, and hence it may be assumed that no gross variation in composition of the individual carbides occurs as result of hardening. This suggests that the observed changes in composition of the extracted carbides as a function of austenitizing temperature (Table 4) are due primarily to a shift in the relative amounts of the three carbides.

* Gulyayev¹⁰ estimates that the free carbon in extracted residues of 18-4-1 high speed steel, austenitized at 2370°F (1300°C), is 0.25 pct of the steel electrolyzed.

METALLOGRAPHIC IDENTIFICATION OF THE CARBIDES

Once the existence of the three carbides in the residues was established, a systematic study was made of their etching characteristics in the solid steel in an effort to identify them under the microscope. Reliable techniques were developed which not only disclosed these carbides positively but rendered it possible to determine their volume percentages by lineal analysis. Diamond dust polishing was necessary to prevent the carbides from standing in relief or from being partly smeared by the softer matrix.

All three carbides are nicely outlined by 1 pct nital or 4 pct picral. This permits the measurement of the total volume percentage of the carbides. After repolishing, the M_6C phase can be etched selectively in a 4 pct aqueous solution of sodium hydroxide saturated with potassium permanganate. The M_6C particles are outlined and stained brown in 4–5 sec. The $M_{23}C_6$ and MC carbides are not attacked. After repolishing once more, the MC carbide is darkened selectively with an elec-

trolytic etch in 1 pct chromic acid, stainless steel being the cathode. In 3–4 sec at about 3 volts, the MC particles are colored grey or black. The M_6C and $M_{23}C_6$ carbides are not attached. Lineal analysis is applied after each of the above etches, and the volume percentage of $M_{23}C_6$ is taken by difference. Proof of the effectiveness of these procedures is presented elsewhere.²⁵

EFFECT OF AUSTENITIZING TEMPERATURE

The pertinent results obtained by lineal analysis on the carbides as polished and by chemical analysis of the carbides as isolated are summarized in Fig 4. For correlation purposes, the percent of retained austenite is also shown.

It is immediately evident that the $M_{23}C_6$ carbide dissolves more readily than the other two on austenitizing, and is practically absent above 2000°F (1090°C). The M_6C carbide dissolves more gradually, while the MC carbide hardly dissolves at all below 2200°F (1205°C). In the customary austen-

itizing range of 2200–2250°F (1205–1235°C), there remains 5–8 vol. pct M_6C and 0.5–1 vol. pct MC. The progressive solution of the $M_{23}C_6$ and M_6C carbides causes an increase in the carbon and alloy contents of the matrix, as shown by the partition curves in Fig 4. These values are calculated from the alloy content of the steel and that of the isolated carbides (Tables 1 and 4). It is interesting to note that most of the chromium in the steel dissolves in the austenite at relatively low temperatures, and this coincides with the decrease in the amount of $M_{23}C_6$ carbide, the amount of M_6C and MC then remaining essentially unchanged. Clearly, $M_{23}C_6$ is a chromium-rich carbide. While the solution of the other elements proceeds during this early stage, signifying that $M_{23}C_6$ also contains these elements, most of the tungsten, molybdenum and vanadium and part of the chromium go into solution above 2000°F. Here the $M_{23}C_6$ has disappeared, the MC remains constant and the M_6C is decreasing. Therefore, the M_6C phase certainly contains tungsten, molybdenum, chromium and vanadium. Other evidence²⁵ not considered here shows that most of the tungsten and molybdenum in the carbides is associated with the M_6C phase, while MC is a vanadium-rich phase.

The curve in Fig 4 showing the partition of carbon between the matrix and the carbides must be regarded as very approximate. The values plotted are admittedly questionable because the carbon analyses of the carbide residues tend to be high in view of the free carbon that precipitates during electrolysis of the quenched samples. The other partition curves in Fig 4 do not suffer from this uncertainty.

Conclusions

1. Two electrolytic methods have been studied for the quantitative isolation of carbides from M-2 high speed steel. The double citrate cell, with continuous filtration of the anolyte, yields quantitative extractions for annealed low alloy as well as high speed steels. This is established on the basis that (a) the theoretical carbon content of cementite is found in the extracted residues of annealed low alloy steels, and (b) the total carbon of these steels and of annealed M-2 steels is accounted for in the residues.

2. The citrate cell possesses the experimental disadvantage, aside from its complexity, that portions of the

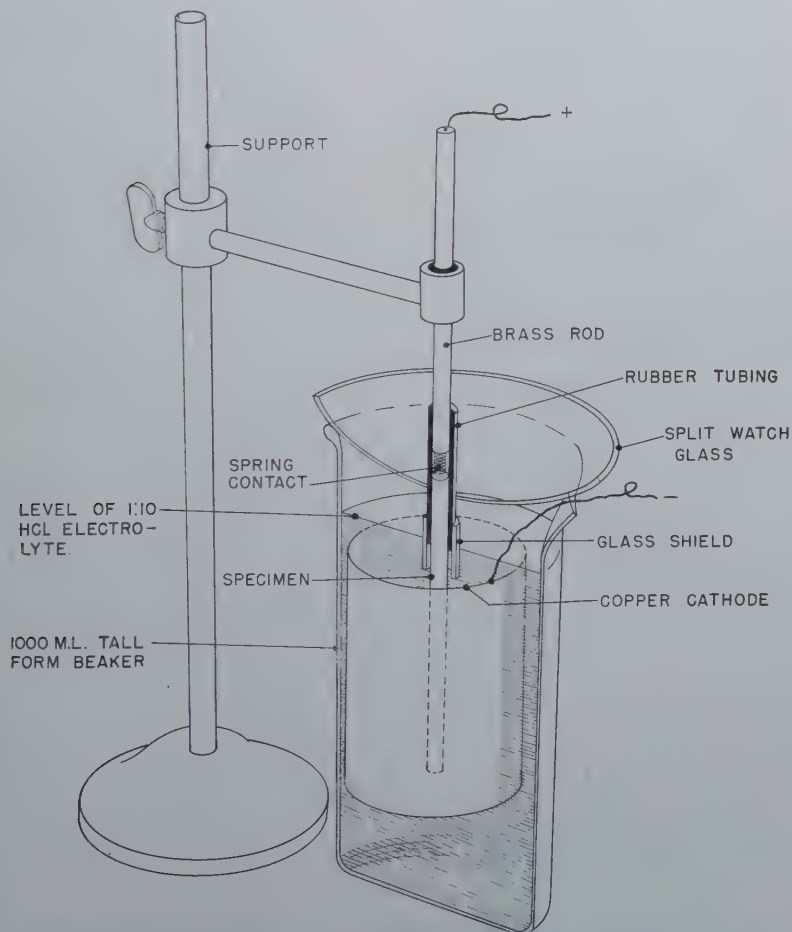


FIG 3—Schematic diagram of hydrochloric acid cell for electrolytic isolation of carbides.
Electrolyte: 1:10 hydrochloric acid.

matrix of high alloy steels may not dissolve during the electrolysis and may thus contaminate the carbide residue. This characteristic becomes more pronounced, the higher the austenitizing temperature. Because the steel particles are ferromagnetic and the carbides are not, it is possible to effect a magnetic separation. However, this procedure is not convenient with the citrate cell.

3. Electrolysis in a simple 1:10 hydrochloric acid cell reduces the extent of the above contamination, and when it does occur, the particles of base metal can be separated reliably with a magnetic method. The hydrochloric acid cell yields quantitative extractions on annealed M-2 steel as shown by the fact that (a) all the carbon in the steel is accounted for in the residue, and (b) the measured densities of the carbide residues are in good agreement with the calculated values based on lineal analysis of the solid steel. The magnetic separation procedure is also quantitative since the total amount of each alloying element in the residue plus electrolyte checks that present originally in the steel.

4. In the electrolysis of hardened M-2 steel, both cells precipitate free carbon from the martensite-austenite matrix. The presence of free carbon lowers the observed density of the carbides extracted from quenched specimens but is not sufficient in weight to have a significant effect on the weight percentage or composition (except for carbon) of the carbides. The carbon values reported for such carbides are undoubtedly high.

5. Three face-centered cubic carbides exist in M-2 steel: M_6C which is based on a complex iron-tungsten-molybdenum carbide, $M_{23}C_6$ which is essentially a chromium-rich carbide, and MC which is a vanadium-rich carbide. $M_{23}C_6$ dissolves completely on austenitizing at temperatures above 2000°F; M_6C dissolves partially, and MC hardly at all. Curves are presented showing the quantitative solution of these carbides and the corresponding partition of the elements between the carbides and the matrix.

Acknowledgments

The authors are grateful to the Vanadium-Alloys Steel Co. for a grant-in-aid to finance this research, for furnishing the steels to be investigated, and for making most of the chemical

analyses. The cooperation of Dr. George A. Roberts, Chief Metallurgist of the above Company, is sincerely appreciated.

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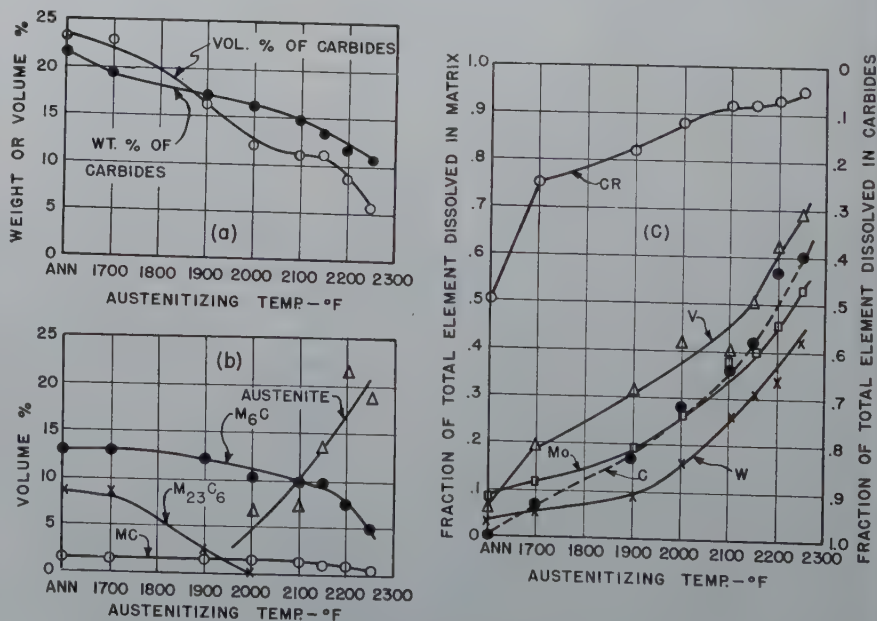


FIG 4—Effect of austenitizing temperature on (a) weight and volume percentages of carbides, (b) amounts of the individual carbides and retained austenite, and (c) partition of elements between carbides and matrix.

M-2 high speed steel (Steel A, Tables 1 and 4).

Solubility of Titanium in Liquid Magnesium

K. T. AUST* and L. M. PIDGEON*

There has been considerable interest in the possible use of titanium in magnesium alloys.¹ Zirconium has shown some promise in this connection² and its general similarity with titanium suggests that the latter might act in a similar manner. A literature survey revealed that quantitative data on the Mg-Ti system was unavailable. Several patents³ have claimed that titanium additions from 0.2 to 4 pct to magnesium alloys were possible, but no mention was made as to the form in which the titanium existed in the alloy. Kroll⁴ succeeded in introducing only traces of titanium into magnesium by bubbling $TiCl_4$ through the metal under argon or by reacting it with sodium titanium fluoride. The application of theoretical data given by Carapella⁵ based on Hume-Rothery's principles, involving atomic size factor, crystal structure, valency and the electro-chemical factor, suggests that a Mg-Ti alloy is a favorable case, and the system appeared to warrant experimental examination.

Experimental Procedure and Results

THERMAL ANALYSIS

If titanium is appreciably soluble in magnesium, a change in the melting point of the magnesium might be detectable using standard cooling curve methods. Magnesium was melted in graphite crucibles under an argon atmosphere, the assembly being enclosed in a silica tube. Graphite thermocouple protection tubes served also to stir the melts. The apparatus was very similar to Fig 1, with the addition of a refractory and baffle system to prevent undue heat losses from the top of the crucible. Chromel-alumel thermocouples were calibrated using Al of 99.97 pct purity.

Dominion Magnesium Limited sup-

Al	Mn	Fe	Ni	Cu	Si	Pb	Ca	Mg
0.007	<0.0005	0.0013	0.001	0.0005	0.0012	0.004	<0.004	99.98 minimum

plied redistilled high purity magnesium of the analysis given above.

Titanium was added in three different forms:

1. Titanium powder -100 mesh, from the Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.

2. Sheet titanium from the U.S. Bureau of Mines, produced by Mg reduction of $TiCl_4$.

3. Magnesium -50 pct titanium master alloy from Metal Hydrides Inc., Beverly, Mass.

The melting point of the high purity magnesium used was measured experimentally as 651.0°C. More than a dozen tests were conducted using titanium from the three sources referred to above, in calculated additions up to 20 pct titanium, at temperatures between the melting point and 1000°C and holding periods up to 6 hr. In no case was evidence obtained of solubility of titanium in magnesium, using inverse-rate and time-temperature curves. The melting point of the magnesium was unchanged within the accuracy of measurement, namely $\pm 0.5^\circ\text{C}$; and no other thermal arrests were detected.

Metallographic investigation of the thermal analysis billets indicated that the titanium additions were apparently mechanically entrapped in the magnesium in segregated areas. Consequently, these samples were not analyzed for titanium. The master alloy proved to be a mechanical mixture of titanium particles in a magne-

sium matrix.

These results indicated that the titanium solubility, if such existed, could not be obtained by the usual thermal methods.

X RAY DIFFRACTION INVESTIGATION

In an effort to detect solubility of titanium in magnesium, samples were investigated using both the Debye-Scherrer and the Focusing Back-Reflection methods. Filings from samples of the thermal analysis billets and from pure magnesium were annealed in argon one hour at 350°C to relieve mechanical strain. Measurements made of the interplanar spacings showed no difference between the Mg-Ti samples and pure magnesium. The interplanar spacings could be measured to within 0.0002Å, and the greatest variation found was 0.0004Å, in the back-reflection method. The diffraction lines for magnesium were not shifted by the titanium additions indicating that the solid solubility of titanium in magnesium is of a very low order—less than 0.5 pct. From both diffraction methods, a d or interplanar spacing of 0.817Å was obtained for the redistilled high purity magnesium. This latter value is not given in the standard X ray diffraction cards for magnesium metal or vacuum distilled magnesium. Theoretical calculations for a close-packed hexagonal space lattice for magnesium indicate that the planes {2134} should give a line which was found. The relative intensity for this reflection at 0.817Å is slightly less than that at 0.870Å for magnesium.

SOLUBILITY OF TITANIUM IN LIQUID MAGNESIUM

The Mg-Mn system was examined by Grogan and Haughton⁶ who were

Cleveland Meeting, October 1949.
TP 2680 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949.
Manuscript received Nov. 18, 1948; revision received May 20, 1949.

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¹ References are at the end of the paper.

and 0.08 pct. These erratic analytical results suggested that finely divided titanium particles, despite the substantial density difference, do not settle readily in liquid magnesium and may not be employed as a source of titanium in liquid solubility tests. Titanium sheet, obtained from the U.S. Bureau of Mines, was used in the remainder of the tests.

Experimental Procedure

The apparatus used for the solubility tests is shown in Fig 1. Each charge consisted of 135 g of the high-purity magnesium and 15 g of titanium sheet. The titanium sheet was wrapped around the graphite thermocouple protection tube and kept in the molten magnesium during the holding periods at the desired temperature. The graphite protection tube also acted as a stirrer during the early portion of each holding period. It was assumed that titanium and graphite would not react to form titanium carbide in any appreciable quantity at the temperatures used in these solubility tests. Cylinder argon of commercial purity was passed through an oil filled bubbler, absorbent glass wool, anhydrous calcium chloride, and a "getter" furnace which contained magnesium chips at 600°C to remove oxygen and nitrogen. The purified argon passed to the system through the steel tube as shown, and escaped to a water trap and bubbler, maintaining a positive pressure of argon in the melt chamber. A chromel-alumel thermocouple was placed in the top part of the melt and the temperature was maintained within $\pm 5^\circ\text{C}$ by an automatic temperature controller.

Sampling

After each holding period, the Ti sheet was lifted from the melt and a steel sampling tube was quickly lowered into the top half of the melt. A liquid sample was pulled into the tube by a static vacuum sufficient to remove samples ranging from 2 to 5 g. After removal from the melt, the steel was stripped from the magnesium, and the entire sample analyzed for titanium except for a very small portion taken for metallographic purposes. Drillings were taken from the top section of the solidified billet to obtain the solubility at the freezing point. The analysis of titanium in magnesium was done colorimetrically using an adaptation of a well-known color reaction.¹² The method was calibrated with a standard titanium solution prepared from ti-

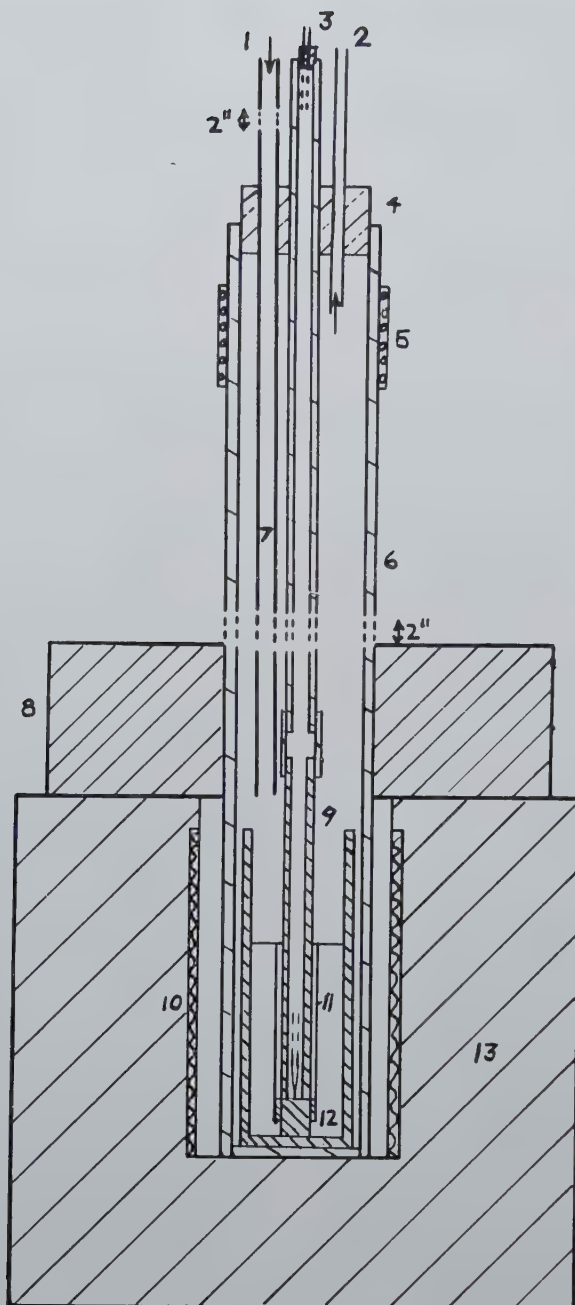


FIG 1—Liquid solubility apparatus.

- | | |
|-------------------------------|--------------------------|
| 1. Argon Inlet | 8. Refractory |
| 2. Argon Outlet | 9. Graphite Thermocouple |
| 3. Thermocouple Tube | 10. Furnace Coil |
| 4. Rubber Stopper | 11. Ti Sheet |
| 5. Water Cooled Cu Coil in Pb | 12. Melt (Mg) |
| 6. Steel Tube | 13. Furnace Refractory |
| 7. Steel Sampling Tube | |

unable to determine the liquidus line by thermal analysis, since it was found impossible to obtain any definite evidence of arrest points. The liquidus line, however, was successfully determined by the analysis of dip samples taken from the top of the melt at definite temperatures. This method has proved satisfactory where the primary insoluble crystals separating from the liquid have a different density from the liquid. A similar technique was used by Fink and Van Horn⁷ to determine the liquidus line for the Al-Ti system and

has also been employed by other investigators.^{8,9,10,11} It was decided to apply this method to the Mg-Ti system.

In preliminary tests, titanium powder was added to liquid magnesium under an argon atmosphere. Analytical results on these dip samples showed gross segregation. For example, titanium analyses of triplicate portions from a test that had been held at 850°C for 8 hr gave 0.012, 0.57 and 0.052 pct, while another group which had been held at 700°C for 8 hr showed 0.28, 0.01

tanium dioxide (NBS Std 154). A blank run of the apparatus with no titanium introduced gave no indication of titanium which, under the conditions of the method, means a content of less than 0.001 pct. None of the other elements present in the magnesium in normal amounts interferes with the analysis.

Experimental Results

If incomplete separation of melt and solid had taken place, the liquid samples as withdrawn might contain some of the solute metal as solid particles. If such had occurred, erratic results would again have appeared, the presence of such an error would be obvious, and those samples would be disregarded. The analytical results obtained on different samples from the same test strongly support the belief that the titanium found was in solution when the sample was taken from the melt (Table 1). The average results are plotted in Fig 2. Fig 3 is a typical photograph taken from the cross-section of the liquid solubility samples. Small dark particles of Ti which have come out of solution on cooling from the sampling temperature to the freezing point may be seen. Magnesium samples without titanium additions did not show this second phase. The even size and distribution of these particles support the belief that they were in solution at the sampling temperature.

Table 1 . . . Experimental Results

Test No.	Temp. °C	Holding Time, Hours	Pct	Titanium
35	651		0.0027	0.0027
46	651		0.0028	0.0023
40	651		0.0043	0.0025
42	651		0.0020	0.0013
42	700	48	0.0062	0.0066
35	775	24	0.011	0.010
40	775	24	0.013	
41	850	24	0.015	0.019
46	850	48	0.015	0.014

Conclusions

1. The solubility of titanium in liquid magnesium is extremely small, increasing approximately from 0.0025 pct at the freezing point of magnesium to 0.015 pct at 850°C. This titanium solubility is far less than that reported for zirconium¹³ or for iron in magnesium.¹⁴

2. Solid solubility of titanium in magnesium could not be detected using X ray diffraction methods.

3. The freezing point of redistilled high-purity magnesium is 651.0°C.

4. An interplanar spacing or d value was obtained for magnesium, 0.817Å, a value which has not been reported in the standard X ray diffraction card index system.

Acknowledgments

The financial assistance received from the Ontario Research Council is gratefully acknowledged by the junior author (K. T. Aust). The authors are indebted to Mr. R. G. Ernst, Research Chemist of the United States Metals Refining Co., N. J., for the titanium analyses, and to Dr. E. J. Allin of the Department of Physics, Toronto, for advice in the X ray analysis.

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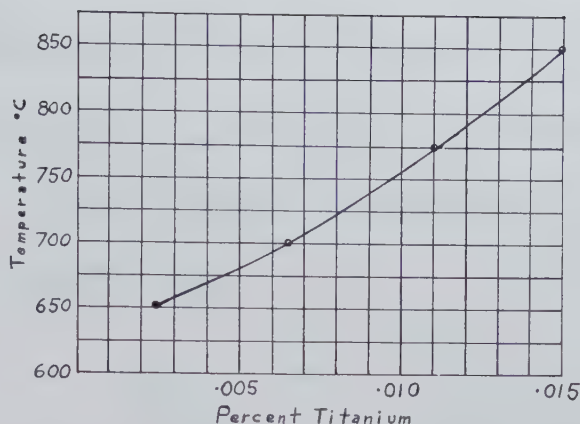


FIG 2—Solubility of Ti in liquid Mg.



FIG 3—Cross-section of typical solubility sample showing precipitated Ti in Mg.

× 150. Etched with 10 pct sodium bicarbonate.

The Lattice Parameters of High Purity Alpha Titanium; and the Effects of Oxygen and Nitrogen on Them*

HOWARD T. CLARK, JR.,† Member AIME

Introduction

Within the last twenty years at least three sets of lattice constants for the room temperature (alpha, hexagonal close packed) phase of titanium have been reported in the literature. These values, summarized in Table 1, differ one from another by amounts far greater than can be explained on the basis of inaccuracies in parameter measurements.

This paper presents new values determined for high purity titanium. It also details the effect of two elements, oxygen and nitrogen, on these parameters and shows that two of the previously reported values are probably in error because of the presence of one or both of these elements in the material used for examination.

Preparation of Samples

The titanium‡ used for this study was prepared by the thermal decomposition of titanium tetraiodide⁷ on a hot titanium filament. This titanium rod was prepared for X ray examination by melting about 10 g in purified argon in an electric arc furnace on a cold hearth. This cast button was cold rolled to 0.040 in. thick sheet, cleaned, annealed in vacuum at 1000°C and cold rolled to 0.020 in. thickness. About 0.003 in. of surface material was then removed by hand grinding on papers to remove any contamination caused by the rolls and the sample was bent to the radius of the focusing camera. The sample was then vacuum annealed at 600°C, a temperature just high enough to cause recrystallization, producing a fine grained structure. Such a structure is essential in sheet

Table 1 . . . Summary of Literature† Values of Alpha Titanium Lattice Parameters

Worker	a_0		c_0		c/a
	KX	Å	KX	Å	
G. Haag ¹	2.945	2.953	4.67	4.729	1.60
J. D. Fast ²	2.945	(2.951)*	4.684	(4.68)*	1.59
Greiner and Ellis ³	2.945	(2.951)	4.684	(4.694)	1.591

* Figures in parentheses have been calculated from the reported KX units for comparison (KX units $\times 1.00203$ = Angstroms).

† Since the preparation of this paper the following values for the lattice parameters of high purity alpha titanium have been published by Bruce W. Gonser (*Metals Progress* (1949) 55, p. 346):

$a_0 = 2.946$ KX units (2.952 Å), $c_0 = 4.686$ KX units (4.695 Å). A comparison of these values with those presented in this paper and with other literature values shown in Table 1 suggests that the X ray samples used by Gonser were contaminated with oxygen and/or nitrogen. This conclusion is based on the fact that his value of a_0 is approximately correct, but that his value of c_0 is extremely high compared with both Fast's value and the writer's, the writer's explanation for which is detailed in this paper.

specimens prepared for focusing cameras in order to have a sufficient quantity of grains to develop continuous diffraction lines.

The use of sheet specimens rather than powder samples appears to be essential for the X ray examination of titanium. This is because of titanium's exceptionally high gettering power and its rapid absorption of adsorbed gases, water vapor, and the like at annealing temperatures. While the effect of this contamination may be neglected for sheet specimens, its effect on 200–300 mesh powder is appreciable.

Titanium alloys of oxygen and nitrogen were prepared by a technique simi-

lar to that described above except that the required amount of the highest purity titanium oxide or nitride available was added prior to melting in the arc furnace. These materials were added by placing them in holes drilled in the titanium. A small weight loss invariably occurs during the melting of these oxygen and nitrogen alloys. This is believed to be due entirely to the loss of some of the powdered compound in the blast of the electric arc, an assumption which is justified on the basis that there is no weight loss during the melting of titanium metal and that wet analyses for nitrogen on representative samples have checked with the calculated analyses when it was assumed that the entire loss during melting was due to the loss of titanium nitride powder. These alloy buttons were reduced to sheet in the same manner as used for the unalloyed titanium.

Experimental Procedure

A North American Phillips 12 cm diam symmetrical precision focusing vacuum camera and copper radiation

Cleveland Meeting, October 1949.
TP 2656 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received April 26, 1949.

* This paper is part of a dissertation to be presented by the author to the Faculty of the Graduate Division of the School of Engineering of Yale University in partial fulfillment of the requirements of the degree of Doctor of Engineering.

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‡ References are at the end of the paper.

‡ This material was prepared by Battelle Memorial Institute under contract for the Remington Arms Co., Inc.

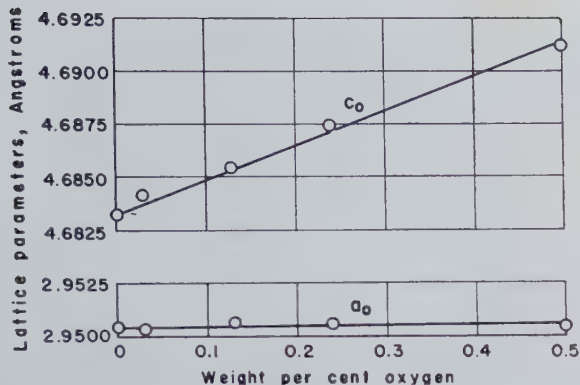


FIG 1—The effect of the addition of small amounts of oxygen on the lattice parameters of alpha titanium.

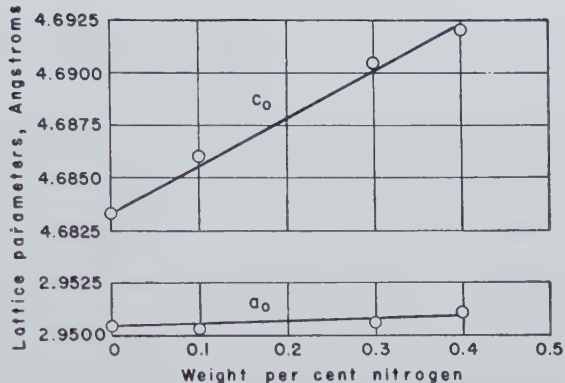


FIG 2—The effect of the addition of small amounts of nitrogen on the lattice parameters of alpha titanium.

were used for all determinations. The films were measured with a conventional measuring unit employing a micrometer screw with a drum graduated in 0.002 mm divisions. Calculations were made using Cohen's⁵ least squares method. The results were analyzed for probable error by the Jette and Foote⁶ method. Table 2 summarizes a typical calculation, the details of which are obvious if reference is made to Cohen's and Jette and Foote's papers.

Table 2 . . . Details of Calculation for Titanium Sample No. 4

Cu $K\alpha_1 = 1.540522 \text{ \AA}$ Cu $K\alpha_2 = 1.544367 \text{ \AA}$
Cu $K\beta_1 = 1.39218 \text{ \AA}$

Line	Plane	Experimental $\sin^2 \theta$	Calculated $\sin^2 \theta$	$\sin^2 \theta \times 10^5$
1	(0006) α_1	0.97399	0.97400	-1
2	(0006) α_2	0.97400	0.97401	-1
3	(3032) α_1	0.92587	0.92592	-5
4	(3032) α_2	0.92592	0.92592	0
5	(1233) α_1	0.87953	0.87939	+14
6	(1233) α_2	0.87950	0.87939	+11
7	(1234) β_1	1.06873*	1.06877	-4
8	(2025) β_1	1.03963	1.03964	-1
9	(3030) α_1	0.81741	0.81752	-11
10	(3030) α_2	0.81753	0.81752	+1
$a_0 = 2.9503 \pm 0.0005^\dagger$				
$c_0 = 4.6827 \pm 0.0004$				
$c/a = 1.587$				

* Values of $\sin^2 \theta$ greater than 1.0 result from the conversion of measured values of $\sin^2 \theta$ to equivalent Cu $K\alpha_1$ values.

† A probability of 0.05 has been taken for determining limits of accuracy.

Room temperature remained fairly constant at about $25 \pm 2^\circ\text{C}$ during these exposures. The camera is so constructed that the synchronous motor used to oscillate the sample does not appreciably increase the sample temperature.

Experimental Results

Table 3 lists the lattice parameters of several different samples prepared from different titanium tetraiodide decomposition runs and reduced to sheet as discussed above. All values for both a_0 and c_0 agree very well.

Oxygen and nitrogen form interstitial solid solutions with alpha titanium, and, as would be expected for interstitial solutes, increase the titanium parameters. The amount of this change produced by each of these elements is shown in Fig 1 and 2 respectively.

Discussion of Results

It will be noted in examining Fig 1 and 2 that the addition of the common contaminants to alpha titanium expands the lattice in the c direction much more than in the a direction. With this in mind, it is interesting to look again at Table 1 and to compare the values presented there with those of Table 3. Such an examination leads to the following conclusions:

1. Fast's values, which were determined on titanium prepared by the same method used for preparing the titanium employed in this investigation, agree with the writer's values.
2. All reported values of a_0 agree within reasonable limits, a condition which is to be expected.
3. The reported values of c_0 vary greatly, the two values reported by Haag and Greiner and Ellis being appreciably higher than the values re-

ported here and by Fast. Greiner and Ellis worked with titanium produced by the Kroll⁴ process which is reported to have small amounts of manganese, magnesium, and silicon present. This material was prepared by powder metallurgy, and undoubtedly also contained oxygen and nitrogen picked up during both the reduction and the leaching and grinding operations, and diffused into the metal during sintering.

Conclusions

1. The lattice constants of alpha titanium are:

$$a_0 = 2.9504 \text{ \AA} \pm 0.0003$$

$$c_0 = 4.6833 \text{ \AA} \pm 0.0003$$

2. The lattice constants of alpha titanium, especially the value of c_0 , are increased appreciably by the addition of small amounts of oxygen or nitrogen.

Acknowledgments

Permission from the Remington Arms Co., Inc. to publish the data in this paper is acknowledged. Assistance was received from several members of the Remington Metals Research Unit, particularly J. A. Snyder on sample preparation, and P. F. Darby on the statistical analysis of the data.

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Table 3 . . . Lattice Constants of Several Different Samples* of Titanium

Sample	a_0	c_0	c/a
1	2.9508 \AA	4.6834 \AA	1.587
2	2.9498 \AA	4.6836 \AA	1.588
3	2.9509 \AA	4.6837 \AA	1.587
4	2.9503 \AA	4.6827 \AA	1.587
average	2.9504 \AA (2.9444 KX)	4.6833 \AA (4.6737 KX)	1.587

* Battelle's best estimate of the chemical composition of these iodide titanium samples is: titanium 99.95 pct, nitrogen 0.002 pct, oxygen undetermined, and carbon, iron, silicon, and aluminum each present in amounts less than 0.03 pct.

The Mechanism of Martensite Formation*

ALDEN B. GRENINGER† and ALEXANDER R. TROIANO,‡ Members AIME

There is need for an adequate working hypothesis that would describe at least qualitatively the crystallographic mechanism for the transformation from austenite to martensite in steel. A general theory would not only be of great assistance in the correlation of existing data and in planning future experimentation, but would also form the basis for an eventual explanation of the property changes that accompany lattice transformations.

The mechanism of martensite formation described in the following pages is the outcome of two new experimental determinations: (1) the accurate evaluation of the lattice relationship between austenite and individual crystals of martensite, and (2) the measurement and analysis of the change in position that a volume of austenite undergoes when it transforms into a crystal of martensite. For this latter study, the stereographic analysis of shear was employed; this method greatly simplifies the solution of lattice-shear problems, and some space is devoted to its description.

Lattice Relationships and Habits

PREVIOUS WORK

The lattice relationships between austenite and martensite in carbon steels have been studied by Kurdjumow and Sachs,¹ and by Wassermann,² and in iron-nickel alloys by Nishiyama,³ Wassermann,² and Mehl and Derge.⁴ The studies by Kurdjumow and Sachs and by Wassermann were made on single (austenite) crystals of quenched 1.4 pct carbon steel, and the results of these two independent studies agree closely with the postulated lattice relationships:

$$\begin{matrix} (111)_\gamma \parallel (011)_\alpha \\ [10\bar{1}]_\gamma \parallel [1\bar{1}1]_\alpha \end{matrix} \quad [1]$$

Both Nishiyama and Wassermann worked with an alloy of iron plus 30 pct nickel (all austenite at room temperature), and "martensitic alpha" was formed by cooling in liquid air. They agree that the lattice relationships for these conditions are:

$$\begin{matrix} (111)_\gamma \parallel (011)_\alpha \\ [11\bar{2}]_\gamma \parallel [01\bar{1}]_\alpha \end{matrix} \quad [2]$$

Mehl and Derge worked with a range of iron-nickel alloy compositions centered about 30 pct nickel. They concluded that when the transformation takes place near room temperature or above, relationship [1] holds, but if the same specimen is made to transform at -195°C , relationship [2] obtains; intermediate temperatures evidently produced a combination of these two relationships. Sachs and co-workers,^{1,7} as well as Nishiyama,³ have proposed transformation mechanisms for martensite formation.* Both of the mechanisms picture the transformation as initiated by shearing movements along the octahedral $[111]$ plane of austenite, followed by suitable expansion

*In all of the above studies, the pole-figure method of evaluating lattice relationships was used. The relationships proposed must be accepted as being close to the correct answer, but not necessarily exact.

A more serious objection to these mechanisms is the relatively large movement and readjustments required. For example, Nishiyama proposes a shear of $19^\circ 28'$ on $(111)_\gamma$ in the direction $[211]_\gamma$, followed by an expansion of 13 pct along $[011]_\gamma$, a contraction of 7.5 pct along $[211]_\gamma$, and a contraction of 1.9 pct in a direction perpendicular to these two directions.

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* This manuscript originally was submitted to the Institute September 27, 1940; and resubmitted without essential change on September 15, 1948. An abstract was published in *Transactions AIME* (1941) 145, 289. The paper was presented at the New York Meeting, February 1941. In view of the increased interest in Transformations, it is now published in its entirety.

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References are at the end of the paper.

and contraction to complete the transformation. These mechanisms have been described and commented upon by Mehl, Barrett, and Smith,⁸ Mehl and Derge,⁴ Wassermann,² and others, and generally accepted by investigators in this field.

Greninger and Troiano⁹ have recently published the results of a detailed study of the shapes and orientation habits of martensite crystals in plain-carbon and nickel steels. They found that, contrary to previous assumptions, martensite plates form not along the octahedral planes but along austenite planes of very high indices; these results were in line with those of previous studies of martensite reactions in nonferrous alloy systems by Greninger and coworkers.^{6,10} Greninger and Troiano concluded that the transformation theories of Kurdjumow and Sachs¹ and of Nishiyama³ are untenable. Phragmén¹¹ reached a similar conclusion.

EXPERIMENTAL METHODS

The object of this particular study was the evaluation of (1) lattice relationship between austenite and martensite, and (2) relationship between the martensite lattice and the martensite plate. The technique used for solving these problems was similar to that used by Greninger⁶ on martensitic structures in β copper-aluminum alloys. Briefly, the method consists of grinding and polishing a specimen on a surface parallel to an individual martensite plate and thus exposing a single martensite crystal for an area of 1 to 3 mm. The orientation of this exposed martensite crystal is then determined by means of a back-reflection Laue X ray pattern.¹² Another pattern is obtained from the matrix crystal, and a stereographic plot of data from these two patterns then provides the solution to the problem. It is necessary to repeat this process on several crystals in order to determine whether or not the solution is unique.

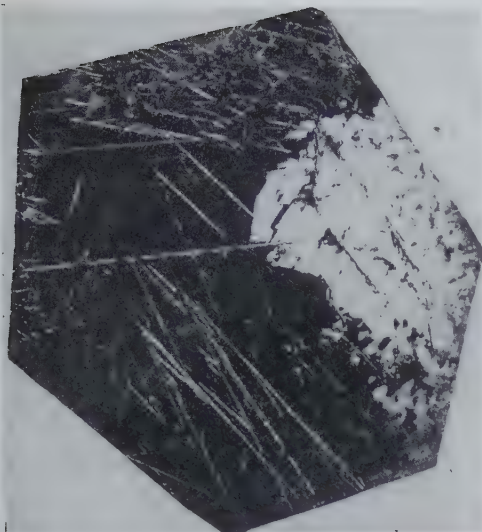


FIG 1—Martensite crystal in nickel steel.

White area is a single crystal of martensite; see text for details of specimen preparation. Etched with Nital. $\times 6$.



FIG 2—Martensite crystal of Fig 1.

Etched with Nital. $\times 1000$.

Most martensite crystals in steel are far too small for the above method to be practical. Judging from past experience, it was decided that the alloy that offered the best chance of success would be a steel containing about 22 pct nickel and 0.8 pct carbon. This alloy is all austenite at room temperature (even after very slow furnace cooling), and small amounts of martensite may be formed by cooling to -77°C . Accordingly, 35 separate 50-g melts of this alloy were prepared in a high-vacuum molybdenum-wound resistance furnace, using as raw materials a 1.3 pct carbon drill-rod steel and nickel shot.* Many ingots were remelted and all melts were solidified very slowly. Resulting austenite grain size averaged about 1 cm. Suitable ingots were cut and ground to form specimens about 1 cm cube, which were then given a metallographic polish on all six surfaces. Each specimen was carefully cooled by successive stages in mixtures of solid CO_2 and acetone; the cooling was halted after a few martensite crystals had formed. The largest martensite plates were about 2 or 3 mm² in area, and about 30 microns thick; most crystals were considerably smaller than this. Suitable specimens were then ground and carefully polished on a surface parallel to a single martensite plate, exposing the crystal for an area of 1 mm² or more. Four martensite crystals from 4 different ingots were prepared in this manner; the largest crystal of this group is illustrated in Fig 1. Back-reflection Laue patterns

were obtained from martensite crystals and the matrix austenite both before and after tempering. The location of the basal plane pole of martensite was checked by means of oscillating crystal X ray patterns.

In addition to the above orientation work, several Debye X ray patterns were prepared from slivers (cut from the ingots utilized) deformed and recrystallized at 1100°C and then cooled to -195°C . Austenite lattice parameter was $3.592 \text{ \AA} \pm 0.005$; martensite parameters were $a_0 = 2.845 \text{ \AA}$, $c/a = 1.045$, both ± 0.005 .

RESULTS

All 4 martensite crystals studied yielded the same solution ($\pm 0.5^{\circ}$); these relationships are expressed in gnomonic projection in Fig 3. It will be noted that the (111) austenite plane is not parallel to the (101) of martensite; the planes are separated by an angle of about 1° . Likewise, no important zones are parallel. Except for the nonparallelism of these two closely packed planes, the evaluated relationship may be described as about midway between the Kurdjumov and Sachs and the Nishiyama relationships. The relationship shown in Fig 3 is for the untempered "tetragonal martensite"; in the process of tempering the relation-

ship changes slightly. The first evidences of tempering are observed in the back-reflection Laue patterns (after only 2 or 3 days at room temperature) by an elongating of the Laue spots on the $[1\bar{1}\bar{1}]$ zone. After a month at room temperature, these spots are considerably elongated, while the spots on both the $[\bar{1}11]$ and $[10\bar{1}]$ zones remain nicely rounded, but shift position slightly. After tempering 2 days at temperatures between 500 and 900°C ,* all spots from the tempered martensite crystal are well rounded, $(111)\gamma$ and $(101)\alpha$ are parallel, and the lattice relationship departs only about 1° from the Nishiyama relationship. Note that the slight change in lattice relationship that occurs during the tempering process (the approximate direction of shift is indicated by the small arrows in Fig 3) is decidedly not in the direction that one would expect from a simple change in axial ratio from 1.045 to 1.

The narrow, dark-etching bands illustrated in Fig 2 were found to be parallel to $\{112\}$; 6 trace normals were located and all of these deviated less than 2° from a $\{112\}$ pole of martensite. The bands are thus probably twin bands, which may have formed during

* After one-half hour at 1000°C , the tempered martensite disappears and the original all-austenite crystal is retained at room temperature; if the austenite is heated to 1100°C , it recrystallizes.

* Most of the ingots were used to prepare specimens for shear studies, described in the second section of this report.

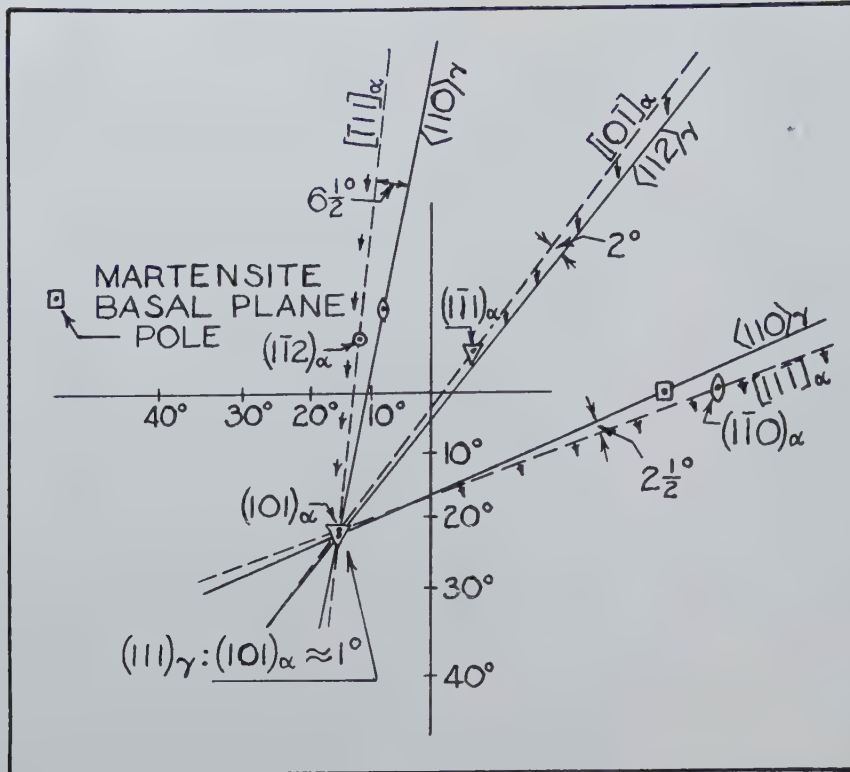


FIG 3—Orientation relationships between austenite and martensite in nickel steel. Relationships are projected gnomonically. Plane of projection is the plane of the martensite plate.

the polishing operation. Individual twin bands seldom are continuous across the midrib of a martensite crystal, and occasionally very small (less than 0.5°) angular differences are observed between bands on opposite sides of the midrib. It is safe to conclude that the midrib plane represents a discontinuity in the lattice of a martensite crystal.

The Martensite Shear*

If a suitable specimen of metastable austenite is polished metallographically and cooled to some subatmospheric temperature to form small amounts of martensite, the martensite may then be seen as a relief effect on the polished surface. This fact has been known for many years, but evidently no one has attempted to measure and analyze this relief.

Fig 4 and 5 illustrate the appearance of the relief formed by martensite in the steel used in the present investigation. A careful inspection of many of these "needles" led us to the conclusion that the relief is caused by a deformation that closely resembles homogeneous shear and that the shear must be in the plane of the martensite plate. Now, it

may readily be proved stereographically that it is impossible to transform a face-centered cubic lattice into a pseudo body-centered tetragonal lattice by a single shear. However, it is possible that two shears are involved in the transformation. If so, and if the first shear can be described by analyzing the martensite relief, then it should be possible to determine the remainder of the transformation mechanism by simple subtraction, inasmuch as the final result of the transformation is known from the lattice relationship and habit information given in Fig 3. Accordingly, attempts were made to analyze the visible martensite shear by methods outlined in Fig 11, Appendix A; the results of this analysis are summarized below.

RESULTS

Twelve martensite crystals in 3 specimens from 3 different ingots were studied. The results obtained for the direction of shear and the shear angle are shown by the small arrows and numbers on the primitive circle of Fig 6; the average γ_0 of the 12 measurements is 10.75° . The accuracy of an individual measurement is about $\pm 10^\circ$ in direction and about $\pm 2^\circ$ in angle. Some measurements were considerably better than this, notably the fifth arrow from the top in Fig 6, which is believed

good to within 5° in direction and 0.5° in angle. If a martensite crystal could be found having a surface like that shown in Fig 4 on two faces of a specimen, the direction could be evaluated to within less than 1° and the angle to within a few minutes. Unfortunately, no near-perfect martensite crystal of this type was found in the dozens of specimens examined, though, of course, only plates that were reasonably well formed were measured. The plates with perfect faces show a definite tendency to be grouped at the centers of the specimen surfaces, instead of at the edges where they must be if they are to be analyzed.

A continuation of the stereographic manipulation yielded the following important result: *When the austenite lattice was deformed stereographically in accordance with the average experimentally determined direction and angle of shear, it was found that a second shear could transform this triclinic lattice into a pseudo body-centered tetragonal lattice of the same orientation and almost the same parameters as martensite (having an atomic volume equal to that of austenite).* Furthermore, this second shear is on the (112) martensite plane in the $[11\bar{1}]$ direction; these are the elements for twin gliding in the body-centered cubic or tetragonal lattice. This second shear cannot, of course, be homogeneous on a macroscopic scale, though atomically it is; it must be confined to the volume of the triclinic lattice produced by the first shear. In the twinning shear of the body-centered cubic lattice, points on every sixth (112) layer are common to both the twinned and the untwinned lattices.¹⁴ In the second shear of the martensite mechanism, the shear angle (the optimum angle is between 12° and 13°) is about one-third as large as the shear angle for twinning, hence the second shear will take place in layers of about 18 atomic planes. The deformation of the austenite lattice into a martensite lattice by means of these two shears is illustrated stereographically in Fig 6,* and the data and results of calculations are summarized in Tables 1 and 2. An inspection of Tables 1 and 2 and of Fig 6 will reveal that the optimum calculated direction for shear No. 1 has been bracketed by directions A and B of Fig 6.

Another possibility for the second shear is that it be on $(123)_\alpha$ in the direction $[11\bar{1}]_\alpha$. These shear elements give

* Readers will find it advisable to examine Appendix A before continuing with this paper. Symbols, equations, and conventions are defined or derived in Appendix A.

* The stereographic portion of this analysis was carried out with the aid of a 15.75 in. diam stereographic net.

slightly better results than (112) and [111] but not sufficiently better to warrant their selection as the true elements. No better results are obtained with irrational shear planes and directions for the second shear.

The missing parameter that makes it impossible at present exactly to define the mechanism from beginning to end concerns the change in atomic volume. When a volume of austenite transforms to martensite, it increases about 4 pct in size; this volume increase is uninfluenced by carbon content (see Table 3). Shear can produce no change in volume; hence, a small but nonetheless important part of the transformation consists of an expansion. If the expansion occurs either at the beginning or end of the transformation, it would produce no change in orientation, and the spacing changes are readily calculated. If the expansion occurs during the transformation, it could have a maximum effect of about 1° on the resulting orientation of the martensite lattice. The "unexpanded martensite" interplanar spacings listed in Tables 1 and 2 were calculated from the measured martensite parameters of the steel used in this study, assuming that the expansion is merely one of a change in atom radius.

Most of the experimental results described above will be discussed in the following section.

The Mechanism of the Martensite Transformation

In the following is presented a brief exposition of the movement of atoms in a martensite transformation. The general statement is really nothing more than a rewording of the experimental results reported in this paper. Application of the theory in a specific case will provide a solution for both lattice relationship and orientation habit for any martensite transformation in steel occurring near room temperature or below. The probable effect of the temperature variable is predicted and it is pointed out that the most important problem awaiting solution is the complete experimental determination of the effect of temperature change on the mechanism of an individual martensite transformation.

GENERAL STATEMENT

In a martensite transformation, billions of atoms move from their positions on the matrix lattice to positions

Table 1 . . . Orientation and Spacing Changes Resulting from the Martensite Shears
Trial Shear "A"

Before Shearing; Austenite Indices	After Shearing; Martensite Indices	Shear No.	Data			Calculated		d-Values† Unexpanded Mart.	Deviation‡ in Degrees
			ρ	ϕ	$\alpha^\dagger$	γ	α'		
(200)	(002)	1	34.°	20.°	1.796	6° 36'	1.645	1.468	1
		2	48.5°	0.°	1.645	5° 12'	1.470		
(022)	(020)	1	153.°	49.5°	1.270	6° 00'	1.331	1.405	1
		2	149.5°	52.°	1.331	6° 22'	1.410		
(022)	(200)	1	136.°	70.°	1.270	2° 02'	1.313	1.405	1
		2	147.°	52.°	1.313	6° 06'	1.396		
(220)	(112)	1	31.°	33.5°	1.270	6° 20'	1.178	1.181	1
		2	No movement	No movement	No movement	No movement	No movement		
(002)	(110)	1	125.5°	0.°	1.796	4° 08'	1.972	1.987	1.3
		2	No movement	No movement	No movement	No movement	No movement		
(111)	(101)	1	63.°	57.5°	2.074	1° 11'	1.990	2.030	0.5
		2	No movement	No movement	No movement	No movement	No movement		
(111)	(011)	1	177.5°	23.°	2.074	10° 12'	2.057	2.030	0.3
		2	No movement	No movement	No movement	No movement	No movement		
(111)	(011)	1	136.°	88.5°	2.074	0° 09'	2.079	2.030	1
		2	4.5°	32.5°	2.079	10° 48'	2.012		
(111)	(101)	1	165.6°	48.°	2.074	7° 11'	2.125	2.030	1
		2	2.°	32.°	2.125	11° 00'	2.072		
(020)	(110)	1	10.°	76.5°	1.796	2° 30'	1.781	1.987	1
		2	135.5°	0.5°	1.781	7° 34'	1.996		

* Refer to Fig 6; shear No. 1 is on the plane of the martensite plate in the direction of Trial Shear "A" with $\gamma = 11^\circ$; shear No. 2 is on (112) α in the direction [111] α with $\gamma = 13^\circ$.

† Calculated from austenite $a_0 = 3.592$ Å.

‡ Calculated from Debye pattern of martensite after changing parameters to make atomic volume equal that of austenite; for unexpanded martensite: $a_0 = 2.810$ Å, $c/a = 1.045$.

§ Angle between the pole resulting from the shear movements and the corresponding martensite pole experimentally located (from the lattice relationship information given in Fig 3).

Table 2 . . . Orientation and Spacing Changes Resulting from the Martensite Shears
Trial Shear "B"

Before Shearing; Austenite Indices	After Shearing; Martensite Indices	Shear No.	Data			Calculated		d-Values† Unexpanded Mart.	Deviation‡ in Degrees
			ρ	ϕ	$\alpha^\dagger$	γ	α_1		
(200)	(002)	1	31.5°	9.°	1.796	7° 02'	1.649	1.468	1.3
		2	50.°	0.°	1.649	4° 51'	1.475		
(022)	(020)	1	153.°	60.°	1.270	4° 22'	1.316	1.405	2.3
		2	150.5°	53.°	1.316	6° 24'	1.390		
(022)	(200)	1	134.°	61.°	1.270	2° 36'	1.328	1.405	1
		2	147.5°	51.°	1.328	6° 20'	1.413		
(112)	(220)	1	31.5°	42.°	1.270	5° 24'	1.192	1.181	0.5
		2	No movement	No movement	No movement	No movement	No movement		

* Refer to Fig 6. Shear No. 1 is on the plane of the martensite plate in the direction of Trial Shear "B" with $\gamma = 10.5^\circ$; shear No. 2 is on (112) α in the direction [111] α with $\gamma = 13^\circ$.

†, ‡, § See Table 1.

Table 3 . . . Atomic Volumes of Austenite and Martensite

Pct Carbon	Temp Range* °C	Atomic Volume Cubic Angstroms per Atom		At. Vol. Mart.† At. Vol. Aust.	Reference
		Austenite	Martensite		
Plain Carbon Steels					
0.90	230° to 20°	11.526	12.058	1.046	Hägg ¹⁶
1.20	160° to 20°	11.664	12.128	1.040	Hägg ¹⁶
1.58	90° to 20°	11.771	12.300	1.045	Hägg ¹⁶
1.74	70° to 20°	11.900	12.450	1.046	Honda and Nishiyama ¹⁷
1.80	65° to 20°	11.928	12.419	1.041	Greninger (unpublished)
Nickel Steel (22 pct nickel)					
~0.80	-60° to -195°	11.586	12.032	1.038	this publication

* Approximate temperature range in which martensite formed. See Ref 5.

† In pure iron, the ratio of the atomic volume of ferrite to that of austenite¹⁹ is: at 900°C, 1.007; at 20°C, 1.039. The average temperature coefficient of expansion of gamma iron is almost twice that of alpha iron. See Ref 18.

on the martensite lattice. This change in position can be described almost entirely by two homogeneous shears (if the transformation cannot be accomplished by one shear, as it can be for the transformation: hexagonal close-packed $\rightleftharpoons$ face-centered cubic). The function of the first shear is to create a

lattice containing a unique set of parallel atomic planes whose interplanar spacing and atom positions are the same as those of a set of parallel atomic planes in the martensite lattice; a second shear, on this unique plane, will then be capable of generating the martensite lattice. In the usual case,

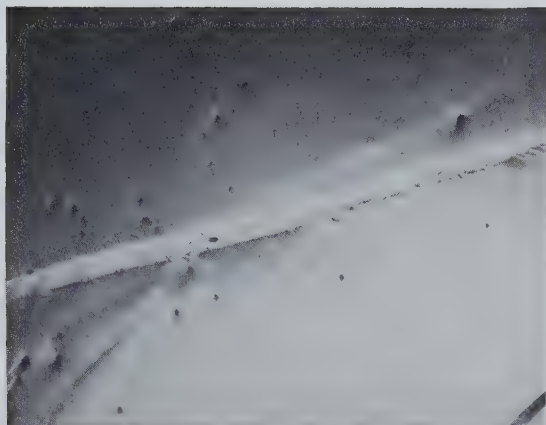


FIG 4—Austenite specimen, polished and cooled to about -77°C .

Unetched. Oblique illumination. $\times 120$. The martensite crystal shown has $\gamma' = 3^{\circ}$ for this surface.

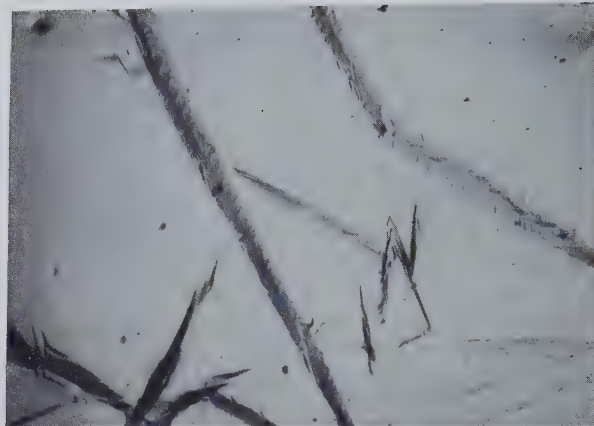


FIG 5—Another area of specimen of Fig 4.

Unetched. Oblique illumination. $\times 120$.

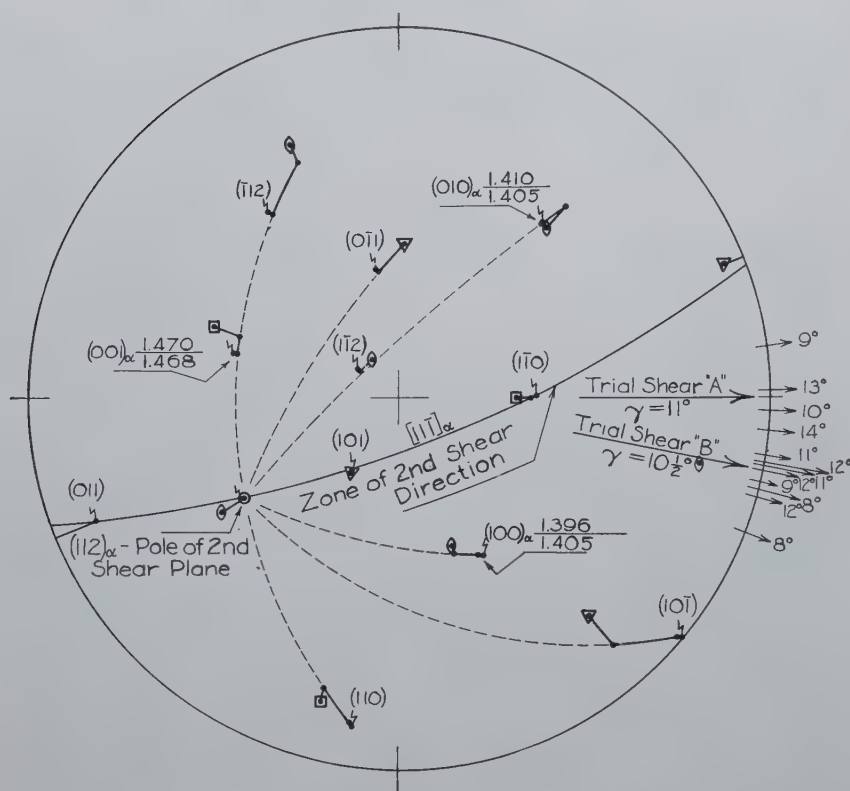


FIG 6—Stereographic projection illustrating the transformation from austenite to martensite by means of two homogeneous shears.

Plane of projection is the plane of the martensite plate (plane of shear No. 1). Small arrows and figures at the right represent the experimentally determined directions and angles of shear for shear No. 1. Trial shears "A" and "B" are directions for shear No. 1 assumed for purposes of calculating orientation and spacing changes of planes in the austenite lattice. Squares, triangles, and ovals designate $\{100\}$, $\{111\}$, and $\{110\}$ austenite poles respectively. Austenite and martensite poles were plotted from experimental data of Fig 3. The travel of each austenite pole as a result of shear No. 1 is along an equatorial line; the additional shift resulting from shear No. 2 is along a great circle joining the pole of the second shear plane (see Tables 1 and 2). The numbers attached to indices of the axial planes of martensite are (1) numerators—interplanar spacings calculated from shear data—and (2) denominators—actual interplanar spacings of the "unexpanded martensite" lattice.

the complete transformation cannot be accomplished *precisely* by two shears; hence a very small readjustment will be needed, most logically after the first shear. The selection of planes and directions for the two shears will be governed by considerations of minimum shear angle and minimum readjustment. The second shear plane will probably be a low-indices plane of the martensite lattice, the first shear plane

(and direction) will be an irrational plane in the matrix lattice because its selection is governed by requirements that cannot be expressed in terms of the matrix lattice itself. The solution of the above problem begins with the selection of the second shear plane.

THE PLANE OF THE SECOND SHEAR

The designation of the second shear plane is arrived at from a comparison of

interplanar spacings of, and atom positions on, the lattice planes of the matrix and martensite lattices; the object is to designate a martensite plane that most easily may be generated from an austenite plane. This comparison must be made with due regard to the principles of lattice shear illustrated in Fig 10, Appendix A. For the transformation face-centered cubic $\rightarrow$ body-centered cubic, the following combinations* are most probable; other possible combinations will have indices higher than those shown:†

- (a) $(220)_{\gamma} \rightarrow (112)_{\alpha}$
- (b) $(331)_{\gamma} \rightarrow (123)_{\alpha}$
- (c) $(442)_{\gamma} \rightarrow (134)_{\alpha}$

If expression (a) can be fulfilled exactly, then expressions (b) and (c) cannot be. Actually, neither (a) nor (b) nor (c) can be fulfilled exactly; the differences in readjustments required by expressions (a) and (b) are so small that for the extension of this analysis, it makes no difference which of the two is selected, or which one actually operates as the second shear plane.

THE PLANE AND DIRECTION OF THE FIRST SHEAR

If $(220)_{\gamma} \rightarrow (112)_{\alpha}$ could be fulfilled exactly, the plane and direction of the first shear for minimum γ could be accurately determined mathematically. Actually, the expression cannot be exactly fulfilled, and the optimum plane and direction of the first shear are more readily located by trial-and-error meth-

* The expression $(220)_{\alpha} \rightarrow (112)_{\gamma}$ means: As a result of a single homogeneous shear in the austenite lattice, the orientation, the interplanar spacing, and the atom positions of the $(220)_{\gamma}$ plane become (very nearly) the same as those of the $(112)_{\alpha}$ plane.

† These expressions (with the direction of the arrows reversed) must also hold for the reverse transformation body-centered cubic $\rightarrow$ face-centered cubic.

ods of stereographic manipulation; of course, there can be no single solution, but instead the shear plane will be located as a small area and the shear direction as a small oriented angle. The spread or scatter for the plane and for the direction will be a function of the readjustments required for the generation of $(112)_\alpha$. For the case of the martensite transformation illustrated in Fig 6, the permissible scatter for the pole of the shear plane was determined stereographically as about $\pm 4^\circ$ and for the shear direction about $\pm 5^\circ$; both of these agreed with those experimentally determined.

LATTICE RELATIONSHIPS

After the elements of the two shears have been determined, the resulting lattice relationship may be solved stereographically. For the transformation face-centered cubic to body-centered cubic, a pseudo body-centered tetragonal lattice of axial ratio greater than 1 will always form; for the reverse transformation, the tetragonal cell must have an axial ratio slightly less than 1 (see Fig 6). The continuation of the transformation to the final cubic form may be hindered by the presence of certain solute atoms;* notably, carbon is responsible for the retention of the tetragonal lattice of martensite in steel. For the martensite transformation in iron-nickel alloys, the tetragonal stage is not retained; we may confidently predict that for nickel contents above about 29 pct† this transformation will proceed along the same paths as the transformation shown in Fig 3, and that the final lattice relationships will be very close to the Nishiyama relationship.‡

THE EFFECT OF TEMPERATURE

It is quite likely that with an increase in the temperature of the transformation, there will be a tendency for the first shear elements to assume lower indices and higher symmetry. However, a change in the elements of the first shear will be possible only when the temperature of the transformation and thus the atomic mobility is increased sufficiently so that larger readjustments are possible. A stereographic

* It is probable that the temporary retention of the tetragonal transition lattice is always due to the proper ordering of solute atoms. The tetragonal lattice that results from the martensite transformation in β brass¹⁰ is remarkably stable; there is good reason to believe that quenched β brass is ordered.¹⁰

† See the first footnote in the next column.

‡ No explanation is advanced for the small lattice-relationship changes observed to occur during the change from tetragonal to cubic martensite, and illustrated in Fig 3.

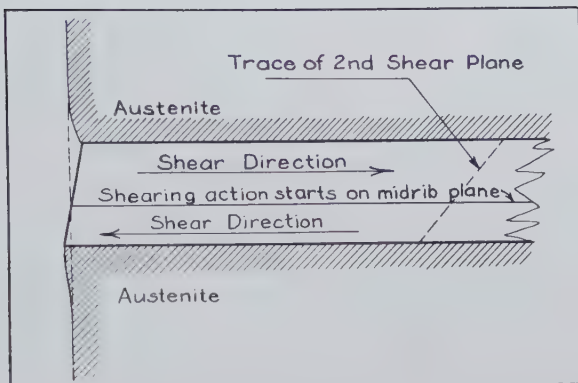


FIG 7—Origin of the martensite midrib.

solution for probable first-shear planes and directions of higher symmetry suggests that the shear plane should become $(422)_\gamma$ and the direction of shear $[\bar{1}11]_\gamma$.* The resulting lattice relationship would then be $[110]_\gamma || [\bar{1}\bar{1}\bar{1}]_\alpha$, and $(111)_\gamma : (101)_\alpha = 0^\circ$ to 3° , depending upon the directions of readjustment; the readjustments necessary are 3 to 5 times as great as those required by the "lowest temperature" mechanism shown in Fig 6. For still higher temperatures, the only probable change in the pole of the first shear plane would be along the $[01\bar{1}]_\gamma$ zone in the direction of $(111)_\gamma$ and for highest temperature, the first shear should be on $(111)_\gamma$ in the direction $[2\bar{1}\bar{1}]_\gamma$ —these are the elements of twinning shear for the face-centered cubic lattice. The above statements are based upon the assumption that the elements of second shear do not change with temperature; there should be no tendency for change, inasmuch as at lowest temperatures, the elements of the second shear are the same as those of the twinning shear in the body-centered cubic lattice. With increasing temperatures, there should be an increasing probability that actual twins will be produced in the body-centered cubic lattice.†

THE MARTENSITE MIDRIB

The plane of the martensite midrib represents a region of discontinuity in

* This predicted shear plane is located only 5° from the experimentally determined plane of martensite plates in plain carbon steel containing 0.9 to 1.4 pct carbon.³ Martensite of this habit is also formed in iron-nickel-carbon alloys in certain composition ranges. Martensite in iron-nickel alloys has the "lowest temperature" irrational habit for nickel contents above about 29 pct nickel; for lower nickel contents the habit is octahedral, but poorly developed.

† Abundant twinning results from the $\gamma \rightarrow \alpha$ transformation in iron; see Ref. 15. For the martensite transformation: body-centered cubic $\rightarrow$ hexagonal close-packed, the plane of second shear is $(10\bar{1}1)$, and polysynthetic twinning on $(10\bar{1}1)$ results from this transformation at moderately low temperatures; see Ref. 6.

the lattice of a martensite crystal.* The manner in which this plane of discontinuity probably originates is illustrated in Fig 7. Shearing action starts on the plane of the midrib, and is propagated in parallel but opposite directions on both sides of this plane. The final tetragonal crystal could be identical and continuous on both sides of the midrib plane if the transformation could be completed without readjustments. The small readjustment that must occur would permit a difference in orientation of about 0.5° , hence a discontinuity at this plane of contact. An additional and probably more important consideration is that it is very unlikely that the shear can be perfect near its plane of origin when it must be propagated on both sides of this plane.

Summary

An X ray and microscopic study of the martensite transformation in steels containing about 22 pct nickel and 0.8 pct carbon has been made, and a brief exposition of the movement of atoms in this martensite-type transformation has been advanced. The martensite crystal is formed from an austenite crystal almost entirely by means of two homogeneous shears. The function of the first shear is to create a lattice containing a unique set of parallel atomic planes whose interplanar spacings and atom positions are the same as those of a set of planes in the martensite lattice; a second shear on this unique plane will then generate the martensite lattice.

The theory predicts both lattice relationships and orientation habits for martensite transformations. The two

* See p. 592. Another fact supports this statement: The midrib plane is the region where carbon first precipitates when martensite is tempered.

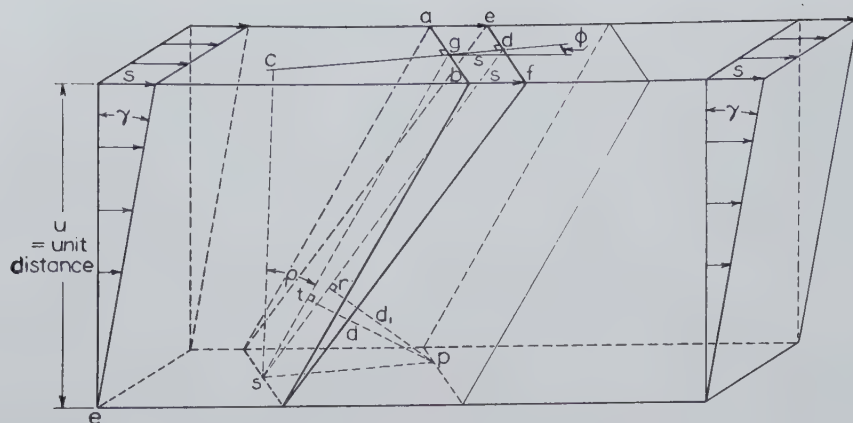


FIG 8—Parallel perspective of homogeneous shear.

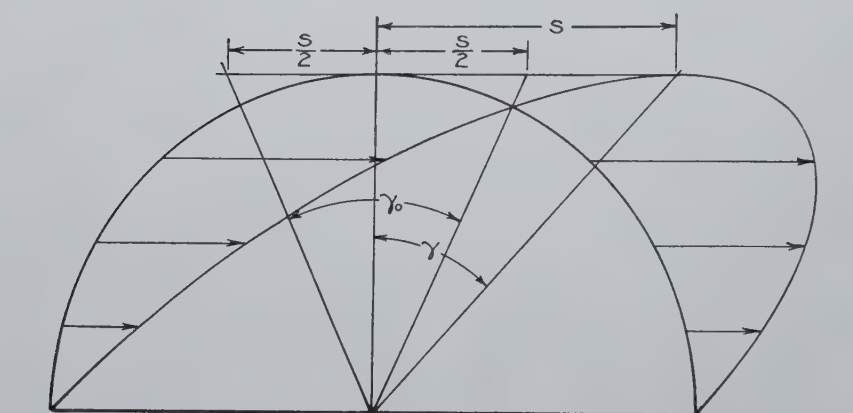


FIG 10—Stereographic summary of shear.
Plane of projection is the plane of shear.

principal experimental determinations were:

1. The evaluation of the lattice relationship between austenite and individual crystals of martensite. (See Fig 3.)
2. The measurement and analysis of the change in position that a volume of austenite undergoes when it transforms into a crystal of martensite (see Fig 6).

Appendix A

The Stereographic Analysis of Shear

In Fig 8 a lattice segment having the shape of a rectangular parallelepiped is deformed by homogeneous shear upon the basal plane in the direction of the long axis. The displacement of any point within the lattice segment is a linear function of its distance from the undistorted base plane. The displacement at unit distance from this base plane is s ; the shear angle is γ . Plane abs is any lattice plane in the undistorted segment; as a result of the deformation, plane abs moves to its new position efs . The position of plane abs is fixed with respect to the plane and direction of shear by the modified spherical polar coordinates ϕ and ρ . ϕ is the angle between the trace normal of plane abs (on the shear plane) and the direction of shear, and ρ is the angle between abs plane normal and the shear plane; ρ is measured in a clockwise direction from the shear plane, and thus may have values between 0° and 180° . The plane $spdgrq$ is perpendicular to planes abs and efs . pt is perpendicular to plane abs , and pr is perpendicular to plane efs . If, then, we designate the angle between planes abs and efs as γ' , the following relations obtain:

$$\tan \gamma = \frac{s}{u}$$

$$\tan \rho = \frac{cg}{u}$$

$$\tan (\rho + \gamma') = \frac{cd}{n}$$

$$gd = s \cos \phi$$

$$\therefore \tan (\rho + \gamma') = \frac{\tan \rho + \tan \gamma}{\cos \phi} \quad [1]$$

In the undistorted lattice, planes parallel to plane abs have interplanar spacing d ; after the deformation, the interplanar spacing of these planes in their new positions is d_1 . Then,

$$\begin{aligned} \cos \rho &= \frac{pq}{ps}, & \cos (\rho + \gamma') &= \frac{pr}{ps} \\ \therefore d_1 &= \frac{d \cos (\rho + \gamma')}{\cos \rho} \quad [2] \end{aligned}$$

All angular distances contained in Eq 1 and 2 are depicted stereographically in Fig 10, in which the plane of projection is the plane of shear and the direction of shear is as indicated. Note that the shear angle γ has been replaced by the quantity γ_0 . (The reason for doing this is that γ_0 is the quantity that is directly determined in the stereographic solution of a shear problem—see Fig 11.) The relation between the shear angle γ and the angle γ_0 is shown in Fig 9. γ_0 is the angle between the circular section of the strain ellipsoid* and the plane that all points on this section occupied in the undistorted lattice; these two surfaces are represented as great circles in Fig 10.

The relationships expressed in Fig 10 are quite general; they may be applied to any case of a single homogeneous shear.† Note that as a result of shear, all poles travel along equatorial lines; the only poles that do not change position are those lying in the zone of the shear direction. The angular displacement, γ' , as well as the change in interplanar spacing may be calculated precisely for any pole that is plotted, so long as the plane and direction of shear and γ_0 are known.‡ The value of γ' and thus of d_1 , are relatively insensitive to small errors in ϕ and ρ .

In the experimental problem that arises, the conditions are the reverse of those outlined above; namely, we measure γ' , ϕ , and ρ for two poles and we wish to solve for the direction of shear and γ_0 . This problem and its stereographic solution are illustrated in Fig 11. In Fig 11, a small plate-shaped volume of the crystal has undergone a deformation as illustrated. Within the distorted volume, surface I has moved to a new position Ia; similarly, surface II is now in position IIa. If we assume that a homogeneous shear along the plane of the plate, we may evaluate the direction and angle of shear. The measurements that must be made are: (1) those ordinarily needed to plot the location of a pole from the measurements of

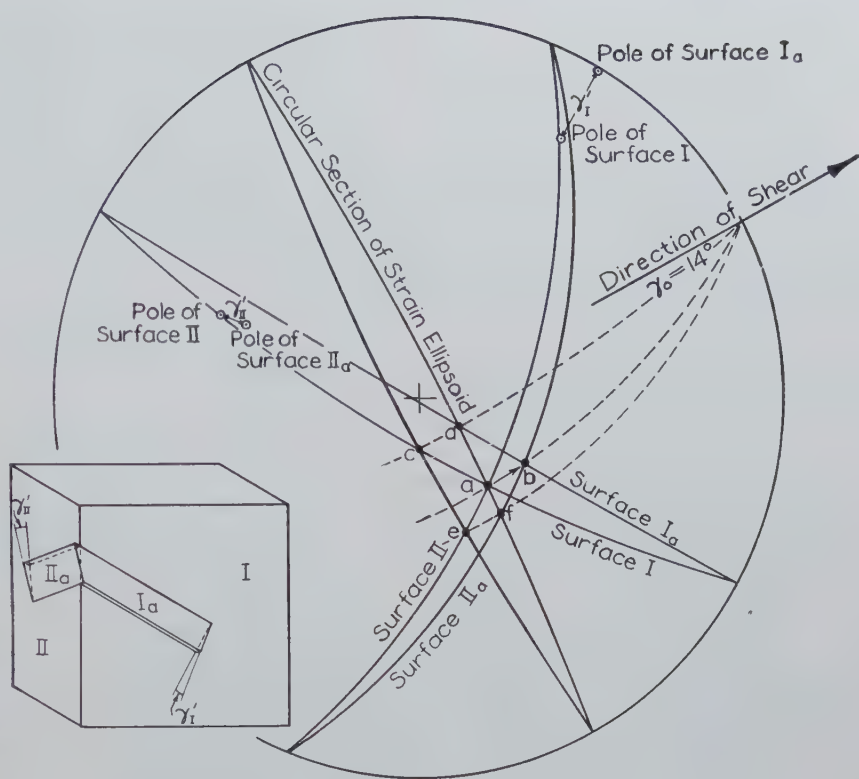


FIG 11—Stereographic determination of the direction and angle of shear. Plane of projection is the plane of shear.

traces of the plane on two surfaces, and (2) angles γ_I' and γ_{II}' ; these latter are measured with an optical goniometer. The steps in the stereographic solution are as follows:

1. With surface I as the plane of projection, plot stereographically the poles of surfaces I, Ia, II, and IIa, and the pole of the shear plane.
2. Rotate the projection to make the shear plane the plane of projection.
3. Replot surfaces I, Ia, II, and IIa as great circles. (This stage is illustrated in Fig 11.)
4. Point a then represents a line in the undistorted crystal (the intersection of surfaces I and II), and as a result of the deformation this line has moved to the position represented by point b (the intersection of surfaces Ia and IIa).

5. The stereographic net is then rotated until a and b lie on the same great circle, and the straight line joining the two diametrically opposite poles of this great circle is parallel to the shear direction. The direction of movement is ascertained from the fact that a moves to b (not b to a), hence the direction of shear indicated by the arrow in Fig 11.

6. To evaluate γ_0 , the stereographic net is rotated until its equator coincides with the direction of shear, and points c and d are located by the requirement

that great circles fad and ec must be at equal distances from the center of the projection and points c and d must lie on the great circle that is shown. Another way of stating the requirement is that the angle between point c and the equator must be equal to the angle between point d and the equator. Angle cd is then equal to γ_0 and angle ef must be equal to cd inasmuch as all points on the great circle ec move to positions on the great circle fad as a result of the deformation.

The stereographic method for solving the above problem represents a graphical solution of two simultaneous equations of form (1). If it is possible to measure γ_I' and γ_{II}' to within a few minutes of arc, and extreme precision is needed, then the shear direction and γ may be solved as follows:

Two equations of form (1), after substituting the known values of γ^1 and ρ for surfaces I and II, reduce to

$$\tan \gamma \cos \phi_I = a \quad [3]$$

$$\tan \gamma \cos \phi_{II} = b \quad [4]$$

divide [3] by [4]

$$\frac{\cos \phi_I}{\cos \phi_{II}} = c \quad [5]$$

then, inasmuch as $(\phi_I + \phi_{II})$ is known, an accurate solution of expression [5] may be obtained by trial and error.

*The other circular section of the strain ellipsoid is, of course, the undistorted base plane. Crystallographers usually designate these two planes as K_2 and K_1 , respectively.

† In the information given by Fig 9, the following space relationships between the hemisphere of projection, the specimen, and the observer are either stated or implied: The hemisphere is located between the specimen and the observer; the shear direction is toward the right of the observer; plane normals proceed from the specimen toward the observer, and intersect the hemisphere of reference in their path; the distortion that is analyzed lies between the undistorted base plane and the hemispherical surface.

‡ The angular displacement may also be determined by stereographic manipulation (see Ref. 13). For most work, however, the stereographic solution of γ' provides insufficient accuracy.

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Effect of Composition on the Wire Textures of Copper and Its Solid Solution Alloys

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It has been proposed¹ on the basis of slip and flow that the ideal deformation texture of drawn wire for face-centered cubic metals is a (111) direction parallel to the wire axis. Under these considerations, an intermediate [100] texture is permissible but should eventually change to the stable [111] orientation. This theory does not explain why copper and its alloys containing up to about 30 pct nickel, 5 pct zinc and 1 pct aluminum were found to have a double [111], [100] texture, while alloys with at least 8 pct zinc and 2 pct aluminum developed essentially a single [111] texture.^{1,2} It does suggest, however, that the [100] component of the former alloys should disappear with additional deformation.³ Some evidence exists to support this suggestion. Howald⁴ reported that annealed copper wire previously drawn 93.8 pct and 98.5 pct developed a single [111] texture. Since, in wire, the drawn and annealed textures are similar, these wires probably had a single [111] deformation texture before annealing. In addition, although cold rolled polycrystalline copper and 80-20 brass were found to form different types of pole figures,^{5,6} single crystals of these metals when rolled from the stable (110) [112] orientation were found to produce essentially the same pole figure.^{7,8} It may be, therefore, that while the addition of solute elements changes the path by which the final

texture is obtained, the final texture is not changed.

Experimental Results

The wires remaining from the previous investigation¹ already drawn about 50 pct reduction in diameter were further drawn to 86 pct and then to 96.4 pct total reduction in diameter. The textures were determined after these stages by transmission X ray photograms taken with a copper K-alpha beam rotated θ degrees toward the wire axis. It was found that the ratio of the [111] and [100] intensities of the double texture in copper and its alloys containing 0.94 pct aluminum, 30.70 pct nickel, 2.61 pct zinc and 4.99 pct zinc increased from about 3:1 for the 50 pct reduction to about 9:1 for the 86 pct reduction. After 96.4 pct reduction, these wires exhibited essentially a single [111] texture.

Summary

Thus, the drawn wire deformation texture of copper and its alpha solid solution alloys containing aluminum,

nickel or zinc is essentially a single [111] texture as predicted by theoretical considerations of slip and flow.¹ For copper and its alloys containing up to about 1 pct aluminum, 5 pct zinc and at least 30 pct nickel, an intermediate [100] component is found after about 50 pct reduction in diameter, but disappears after about 95 pct reduction in diameter. For alpha solid solution alloys containing at least about 2 pct aluminum and 8 pct zinc the single [111] texture is developed considerably more easily and is found after about 50 pct reduction in diameter. No explanation of this compositional effect is apparent. Similar work is contemplated for cold rolled and cold compressed alloys.

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Technical Note No. 19 E. Manuscript received May 12, 1949.

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¹ References are at the end of the note.

Structure and Nature of Kink Bands in Zinc*

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Single crystal rods of cadmium collapse under uniaxial compression into peculiar kinks if the (0001) glide plane is nearly parallel to the axis of compression. In his report first describing this behavior, Orowan¹ proposed that these "kink bands" do not develop entirely by ordinary gliding and twinning but by a special mechanism in which glide lamellae of uniform thickness snap abruptly to a tilted position that permits lattice continuity across the glide planes. He concluded that a portion of each boundary of a kink band consists of a plane (non-crystallographic) which bisects the angle between the glide planes on the two sides of it, and along which the bending and the dislocations are concentrated.

The data presented by Orowan seemed to us to be insufficient to conclude that a wholly new mechanism of deformation was responsible for the kinks. Therefore, we have investigated zinc, which is hexagonal close-packed and has an axial ratio near to cadmium, to see whether similar bands are produced, and whether the ordinary mechanisms of glide, bend-gliding, twinning, and the formation of deformation bands can account for its mode of deformation.

We find that kinks form profusely in zinc as shown in Fig 1. The structure of these kinks, reported below, seems to require no new mechanism for their explanation, but indicates that kink bands are a special form of deformation bands and develop gradually, solely from basal glide. A theory covering the formation and structure of the bands is presented in detail because it is seldom possible to study deformation bands under such simple conditions, yet many principles applying to these kinks

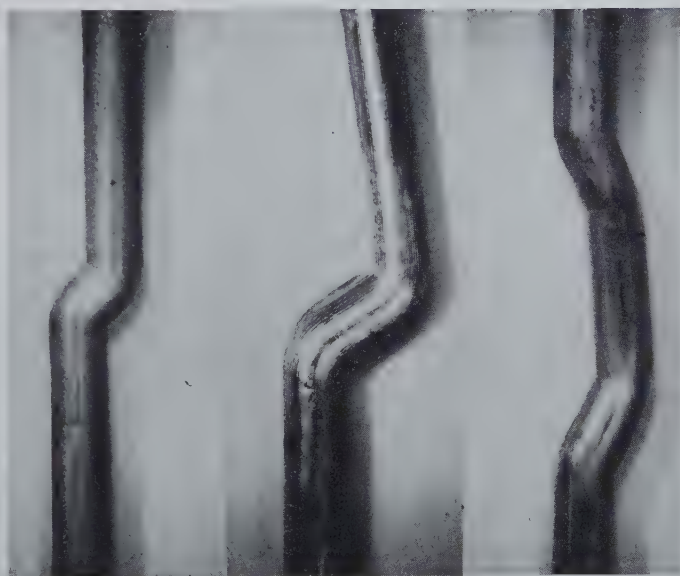


FIG 1—Typical kink bands in compressed single-crystal zinc rod. 3X.

should also apply to deformation bands in general.

Experimental Procedures

Single crystal rods of 2–3 mm diam and 10–15 cm length were grown by the Bridgman technique from swaged wire of Horsehead Special zinc in uncoated pyrex tubes. Orientations of the rods were determined with an optical goniometer after cleaving the rods at

liquid air temperature. Crystals of desired orientations were then selected and used as "seeds" to grow additional crystals approximately 4 ft in length from which all specimens were cut.

The wires were compressed in two ways: in a simple hydraulic handpress—a "soft" machine—and in a Riehle hydraulic testing machine modified to produce very "stiff" action by the simple expedient of inserting lengths of steel bars at the sides of the zinc crystals between the compression plates.

For metallographic examination, the specimens were mounted in sealing wax. Hand polishing was employed, with no grits coarser than No. 1 paper; following the abrasives about 0.025 in. was removed by an etch-polishing reagent.* Final etching to reveal ori-

* 200 g CrO₃, 15 g Na₂SO₄ (or 34 g Na₂SO₄·10H₂O), 1000 ml water, applied by swabbing.

Cleveland Meeting, October 1949.
TP 2688 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received May 2, 1949.

* This research was supported in part by the Office of Naval Research, USN (Contract N-6ori-20-IV).

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¹ References are at the end of the paper.

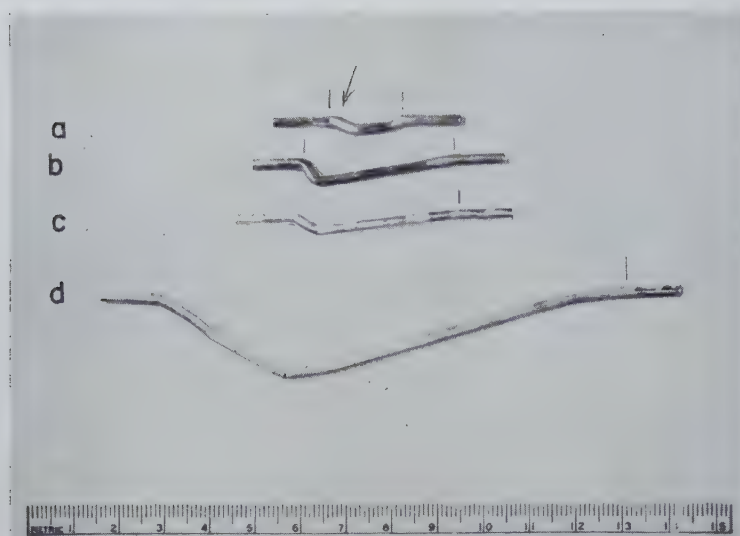


FIG 2—End-kinked crystals, illustrating the increase in kink length with increasing specimen length.

The unsupported length is indicated. The first kink to form in the shortest specimen is shown by the arrow. Specimens *a* and *b* are shown at higher magnification in Fig 1b and 1c respectively.

entations was with 50 pct HCl,² swabbed for about 5 sec. An optical goniometer was used for orientation determinations.

Experimental Results

CONDITIONS FOR KINK FORMATION

A single crystal zinc rod of suitable orientation deforms by kinking when it is compressed axially provided the rod ends are restrained from rotating. Such restraint is provided by inserting the ends into axially aligned holes in the compression plates, or, with short specimens, merely by permitting the squared-off ends of the crystals to rest directly upon the compression plates.

Axial compression alone is not sufficient to cause kinking, for when the ends of a zinc rod are inserted into hardened steel caps having axially-aligned conical points which rest against the compression plates, kinking does not result. Instead, the crystals buckle into a smooth sine-curve without abrupt bend angles on either the convex or concave surfaces. Continuing compression under these conditions leads to twinning at the convex (tension) surface and ultimately to cleavage on the new basal planes of the twinned region; the fracture is then propagated jerkily across the section by the process of further (10 $\bar{1}$ 2) twinning at the base of the crack and basal cleavage through these twins, and the final cleavage surface has a striated, stepped appearance in units corresponding to the widths of the succes-

sive twin bands. Crystals subjected to pure bending without simultaneous compression deform and cleave in like manner.

All crystals having their basal (0001) planes nearly parallel to the wire axis could be made to kink. As the inclination of the basal plane to the wire axis increased, kinking became erratic, so the present work was done on crystals having the basal plane within 3° of the wire axis. The appearance of the kink bands in zinc, Fig 1, 2, and 5 are identical to the photographs of kinks in cadmium published by Orowan.

The kinks occur at several reasonably definite locations along the specimen lengths. For example, in one group of 14 specimens of widely differing lengths, the first kink to appear in 5 specimens occurred at the end of the unsupported length, that is, where the rod entered the hole in the compression plate; in 5 others the first kink formed $\frac{1}{3}$ of the way along the unsupported length, and the remaining 4 kinked at $\frac{1}{4}$ -length positions. Invariably the kinks that formed at the ends formed only in those specimens whose surfaces appeared to be completely free of pits, small laps, or other defects. Conversely, the $\frac{1}{3}$ and $\frac{1}{4}$ locations occurred in specimens containing such blemishes, and, with only few exceptions, the kinked regions were observed to terminate at such visible defects.

There was no correlation between specimen length and kink location. However, in the case of end-kinked specimens, there appeared to be a direct relation between the specimen length and the length of the kink that

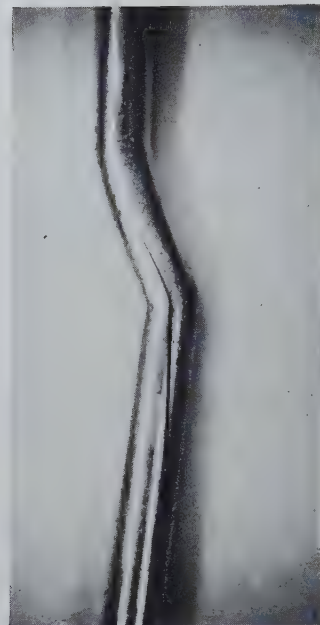


FIG 3—A kinked crystal subsequently cleaved on the basal plane. 3X.

formed—the longer the specimen the longer the kink, as illustrated in Fig 2, and equal length specimens yielded equal length kinks. Furthermore, whenever a second kink formed in such specimens, the new kink was always shorter than the initial kink and always tilted the opposite way. An example is shown in Fig 1c.

The sudden buckling discussed by Orowan is characteristic only of kinking in a “soft” machine; in a “stiff” machine the kinks form gradually, with progressive reorientation of the crystal within the kink bands. For a typical example, a simple kink such as shown in Fig 1a was found to have the basal planes bent 12.5° at the kink notches when it was first unmistakably recognized, but continued compression in a “stiff” machine increased these angles gradually to 27.5° before the test was halted. Final bend angles as large as 88° have been measured.

REORIENTATION IN SIMPLE KINK BANDS

The reorientation caused by kinking is a rotation around an axis that lies parallel to the glide planes. This fact is demonstrated convincingly by cleaving a kinked crystal at liquid air temperature so that the bends and curvatures of the glide planes are revealed, as shown in Fig 3. During the initial stages of kinking the rotation axis is normal to the [2 $\bar{1}$ 10] slip direction that lies closest to the compression axis. In the late stages the rotation axes

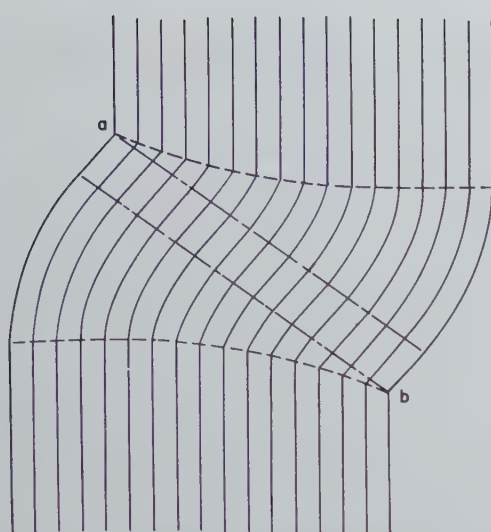


FIG 4—Structure of typical simple kink.

Thin parallel lines indicate glide planes; broken lines, kink boundaries; dash-dotted lines, undistorted parallelogram.

deviate slightly from this crystallographic direction to become non-crystallographic and more nearly normal to the rod axis, though still remaining parallel to the glide planes. For the special case in which a $[2\bar{1}10]$ direction is parallel to the rod axis, the rotation axis does not shift from a position that is normal to both the rod axis and the slip direction.

It is simpler to consider the latter case and to represent the orientations present by a sketch in which the rotation axis is normal to the plane of the drawing. This is done in Fig 4 for a simple kink. It will be noted that on the cross-section of the specimen illustrated in this figure there are two symmetrical areas of oppositely bent glide planes, between which there is a parallelogram of unbent, but rotated, material. The parallelogram has diametrically opposite corners at the kink grooves *a* and *b*. At an earlier stage in the development of a simple kink such as this, the grooves are at the same places and the parallelogram is wider, with sides extending across from grooves *a* and *b* more nearly perpendicular to the wire axis, and with the orientation of material within the parallelogram more nearly like that of the original undeformed crystal. At a later stage in the compression of such a crystal the sides of the parallelogram may meet, leaving no unbent area. When this happens the loci of those parts of the crystal that have been rotated the greatest amount then lie along the line joining the grooves.

In the regions of a simple kink band neighboring the parallelogram of un-

distorted material, the bent glide planes approach the form of arcs of concentric cylinders, with their axes of curvature at the grooves on the surface, as indicated in Fig 4. However, where the arcs approach junction with the undeformed matrix, their radii of curvature decrease progressively, with the centers of curvature for these decreasing radii lying in the two symmetrical boundaries of the kink band. The bending of the glide planes may be considered a special case of the "bend-gliding" studied by the early investigators of crystal plasticity.

KINK BAND BOUNDARIES

The abrupt bends in the glide planes at the boundaries of a kink band, which are the loci of centers of the various radii of curvature mentioned above, mark out a pair of surfaces which are slightly but definitely curved and nearly normal to the axis of the wire at the convex side. The angle of abrupt bending at these boundaries is a maximum at the groove on the surface and decreases gradually across the specimen toward the convex side, corresponding to the changes in radii of curvature.

To study the structure of a kink at the very earliest stage of its formation, a wire was coated with diluted Canada Balsam cement and dried for several days at room temperature. The coated specimen was then compressed in small increments and examined microscopi-

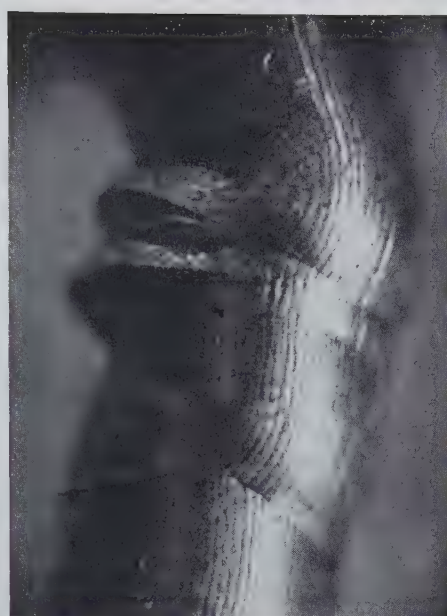


FIG 5—Macrograph of ridges produced in late stage of kinking.

(The striations are due to the method of preparing the single crystals, but in this example correspond to the traces of the (0001) planes as well). 11 $\times$.

cally after each increment until two small cracks were detected in the brittle cement coating. At this stage no macroscopic bend was measurable, but metallographic examination of the cross-section at the cracks revealed a single pair of boundaries, approximately normal to the specimen axis, where abrupt bending through a slight angle occurred. The boundaries extended entirely across the specimen with uniform (though slight) contrast, differing in this respect from the later stage of Fig 4, where contrast increases on approaching the grooves. The material between the boundaries appeared to be unbent, though slightly rotated.

The abrupt boundaries of a simple kink are fixed in the earliest detectible stages of deformation and do not wander through the specimen as deformation proceeds.

SECONDARY BOUNDARIES AND RIDGES

A number of complexities were noted. Additional faint boundaries of abrupt orientation changes were frequently detected. These boundaries occurred either as branches from the primary or other secondary boundaries, or as new and independent boundaries. Their low contrast indicated that their abrupt bend angles were small.

Ridges like those shown in Fig 5 and sketched in Fig 6 often develop in the later stages. Goniometric measure-

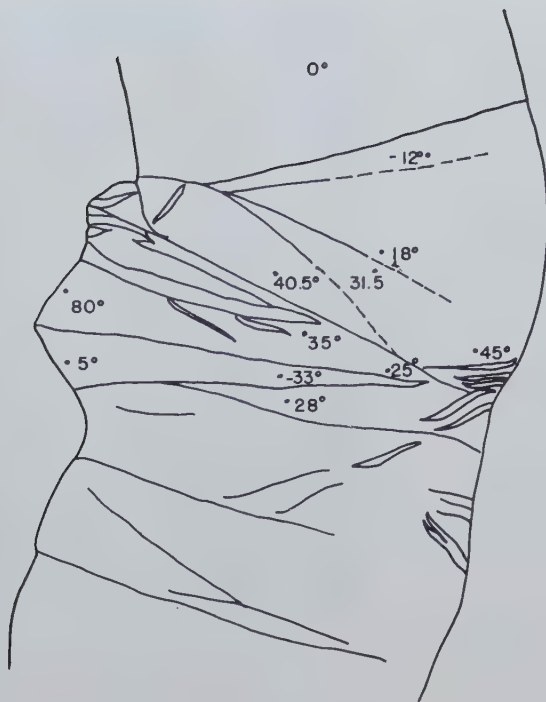


FIG 6—Structure of complex kink of Fig 5.

Numbers at the dots indicate the rotation of the glide planes from their original orientation (0°) parallel to the rod axis. Abrupt bending of the glide planes occurs across the boundaries shown as thin solid lines. Overlapping segments and varying orientations within a segment are seen.

ments such as those recorded in Fig 6 indicate that the ridges are at the boundaries of segments that are rotated in opposite direction, an amount that increases with the amount of deformation. In one example, the bend at a boundary reached 110° . The ridges form on the curved surfaces of prior simple kink bands only after several of these have formed elsewhere along the specimen, and when the local stress conditions must have been greatly altered. They form on the side of the specimen where the compressive strain is the greatest. Branched, intersecting, and overlapping ridges are not uncommon. The ridge and segment boundaries do not, in general, extend across the entire cross-section.

Deformation twinning does not appear to be involved in any manner in the sequence of events of kinking. While there were occasional twins pre-existing in some of the specimens, these did not appear either to initiate or to inhibit kinking. Orientation measurements in the segments at the ridges indicate that these are not twins; the directions of their boundaries are incompatible with twinning, as is also the fact that the ridge angle becomes gradually more acute with continued deformation.

Discussion of Results

The characteristics of kink bands in zinc (and we believe in cadmium also) allow them to be classified as a special form of deformation bands. Deformation bands are lamellar regions in crystals and polycrystalline grains within which there has been reorientation by rotation as a result of slip, the amount of rotation increasing gradually with increasing strain. There are abrupt changes in orientation at the boundaries of deformation bands, and fre-

quently there is also bending (bend-gliding) within the bands. While deformation bands have apparently not been reported previously in zinc, they are a common mechanism of deformation in many cubic metals,³ and no principle is known that would indicate that they should not occur in zinc, cadmium, or other hexagonal crystals provided the applied stress does not first cause twinning. Since compressive stress directed along the basal plane is required for kink bands, they can be expected only in those crystals that are not subject to twinning from this type of stressing, hence only in hexagonal metals or alloys having an axial ratio c/a greater than 1.732.

Actually, nearly analogous structures have been reported by R. F. Miller^{2,4} near the grips in zinc single crystals elongated in tension. Miller identified a "bend-plane"—the locus of abrupt bend angles in the basal plane—and showed that it was non-crystallographic and that its orientation was dependent upon the amount of deformation. However, he described the phenomenon as a new type of twinning, and failed to recognize the structures as deformation bands.

Orowan's suggestion¹ that a new mechanism of deformation is involved in the formation of kink bands seems unnecessary, at least in zinc, since the structure and formation of the bands can be accounted for by the accepted mechanisms of slip and bend-gliding that operate in forming deformation bands. Contrary to Orowan's conclusions regarding kinking in cadmium, the glide planes do not snap over suddenly to the kinked orientation, nor is there a " k plane"¹ extending from the grooves to the center of the wire over which there is a constant bend angle equal to the bend angle at the groove.

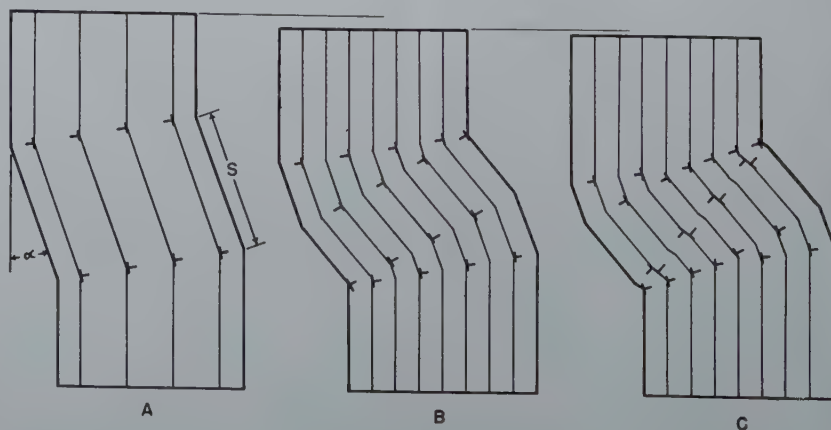


FIG 7—Formation of a kink by dislocations.

Each dislocation is marked by a T with the extra plane of atom indicated by the stem of the T. Successive stages are shown in drawings A, B and C.

However, we are in agreement with Orowan's suggestion that there are dislocations concentrated in the boundaries, and we develop in the following section a detailed qualitative theory of the bands based on the formation and movement of pairs of dislocations.

The fact that there is an axis of rotation lying in the glide plane of zinc and parallel to the boundaries of the band, suggests that rotation axes may be similarly oriented in deformation bands of other metals, but observations on this point seem to be lacking. The rotation axis should be crystallographic and lie in the glide plane normal to the glide direction when a single slip system operates, but could well have irrational indices if two glide systems are active. A single slip system appears to be active in zinc during the initial stage of kinking, because the rotation axis $[01\bar{1}0]$ is exactly that predicted for such behavior.

In a crystal oriented with the glide direction parallel to the axis of compression, the rotation is such as to keep the maximum resolved shear stress in the glide plane continually parallel to the glide direction. Wandering of the maximum resolved shear stress direction occurs, however, in less symmetrically oriented crystals and may ultimately induce glide in a second glide direction, with a corresponding change in the axis of bending of the glide planes. If two glide directions are at equal angles to the axis of compression initially, they would be expected to remain so throughout, and bending would then be around $[1\bar{2}10]$. Such behavior was confirmed experimentally in most specimens of this orientation; however, a few did exhibit less symmetrical rotation and had axes with irrational indices. In these exceptional cases the behavior was not consistent throughout the region of a single kink,

suggesting that defects are possible in a glide plane which do not affect all slip directions equally.

A DISLOCATION THEORY OF KINK BANDS

The development of a kink band can readily be described in terms of dislocations. Initial plastic flow in these specially oriented zinc crystals appears to be confined to the volume of the kink band and does not extend to the ends of the specimen. From this and considerations discussed below we conclude that flow begins by the generation, within the volume that is to become the band, of pairs of dislocations of opposite sign, the positive and negative dislocations collecting, respectively, in two parallel planes that are nearly perpendicular to the glide planes.

The generation of such pairs of dislocations may be expected from consideration of the elastic distortions produced in slender rods by compressive loading. According to the theory of elasticity,⁵ an isotropic slender rod with fixed ends buckles in the shape of one full period of a cosine function, the ends being tangent to the wave at successive crests. If the anisotropic zinc crystals of the present experiments are assumed to buckle elastically in similar cosine shape, then this distortion produces a shear stress along all the basal planes, the magnitude of which is a sine function of position along the rod length and is proportional to the amplitude of the cosine flexure. Shear stress maxima consequently occur at the points of inflection of the cosine flexure, the quarter-length locations. Further decrease of the distance between the compression plates increases the amplitude of the cosine flexure, and hence increases the shear stresses. At

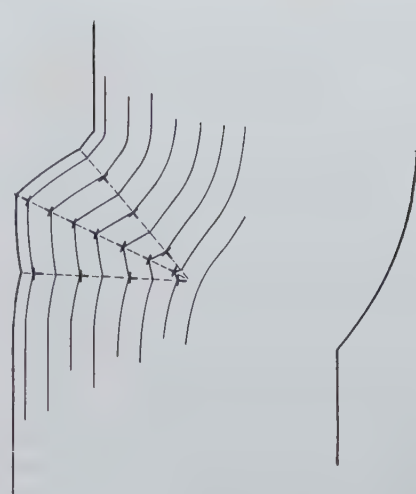


FIG 9—Formation of a ridge by dislocations.

some critical shear stress a pair of dislocations of opposite signs might be generated, either as linear dislocations, or more probably as screw dislocations spreading out from some point, possibly at the surface, and becoming a pair of linear dislocations. Since the critical shear stress is attained on all planes simultaneously but is localized to a short length of area on each, its relief must entail the generation of pairs of dislocations on many separate planes approximately evenly spaced over the cross-section rather than the generation of an equal number of pairs on one, or only a few, planes. We conclude that the positive and negative dislocations must collect, respectively, into planar arrays because, if they did not, there would be additional local stresses set up around them that would increase the strain energy of the structure.

The result of such a pair of planar arrays of dislocations of opposite signs is a slight bend of the glide planes at one of the planes of dislocations and an equal reverse bend at the opposite plane of dislocations. This bend is illustrated in Fig 7a for the simplest case where a slip direction is initially parallel to the rod axis. Each plane of dislocations bisects the angle between the glide planes on the two sides of it because, if it did not, the interplanar spacings of the glide planes could not be equal on both sides of the bend angle, and hence the strain energy of the configuration would be increased. Because the symmetrically oriented planar array is a configuration of minimum strain energy for a given bend angle, individual dislocations that depart from it will be forced back into it. Strain energy considerations also

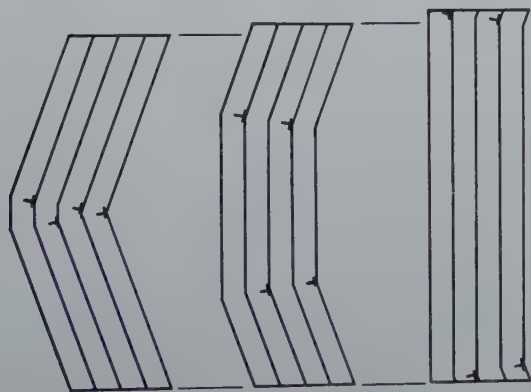


FIG 8—Tensile strain by the separation of like planar arrays of dislocations.

lead to the conclusion that dislocations are approximately evenly spaced over these planes, from one side of the wire across to the other side, because if this were not so some portions of the crystal would be bent more than others and an additional strain energy would be introduced. Local lattice imperfections and stress raisers might distort the regular array of dislocations on a local (atomic) scale, but on the average there should be a remarkably uniform distribution over the planes.

The planes of dislocations are forced apart by the applied stress and thereby shorten the distance between the compression plates. The possibility of deformation by the movement of such arrays of dislocations has previously been proposed by Bragg.⁶ For the case sketched in Fig 7a the shortening is given by the formula $s(1 - \cos \alpha)$ where s is the distance between the planes of dislocations, and α is the bend angle at each. This angle is proportional to the number of dislocations in the arrays.

The planes of dislocations move apart until they are stopped by some agency, discussed below, where they become the permanent boundaries of the kink band. Further compressive strain occurs not by increasing the spacing between the boundaries but by increasing the rotation, that is, by generating new pairs of dislocations. Since the glide planes within the band are now rotated to a position where they have a higher resolved shear stress than the glide planes in the matrix outside of the band, the new pairs will be generated within the band.

The new dislocations array themselves on a new pair of planes, the position of minimum energy for them. These planes begin to separate as the first pair did, and continue until they encounter the first set, as illustrated in Fig 7b. Since each of the second pair of planes bisects the glide planes on each side of it, and since the second pair is generated in glide planes that have already been reoriented by the first pair, it follows that the second pair will be tilted with respect to the first pair in the manner illustrated (in an exaggerated way) in the sketch, and the first contact of the second pair with the first pair will be at the grooves. As successive pairs are generated (Fig 7c), the bend angle at the groove will steadily increase. If each pair has about the same number of dislocations in it, the result of many successive pairs of planes will be a bending of the

glide planes into concentric cylinders with the axis of the cylinders centered at the grooves.

However, if the movement of all successive planes of dislocations were stopped by initial contact with the first planes as sketched in Fig 7b, there would be no visible grooves extending nearly half-way around the rod circumference such as is observed experimentally. Instead it appears that the movement of the second pair of dislocation planes is not stopped immediately by contact with the first pair at the groove locations, but is merged with the first set over part of its area, increasing the uniform bend angle over the coalesced area and producing bending elsewhere as sketched in Fig 7c. Then if succeeding pairs contact the boundary set along progressively decreasing areas following their encounters at the grooves, the final pattern of dislocations will yield the orientations sketched in Fig 4. The boundaries will be slightly curved, as indicated in Fig 4 and 7c, and the minimum strain energy argument would suggest that the boundaries be curved so as to bisect the abrupt bend angle of the glide planes at all points.

Reasoning from the minimum strain energy argument, we would expect complete merging of two arrays of dislocations that have contacted each other at a point, because near the contact region there would be local distortions on an atomic scale from the close approach of the arrays that would not be required for a coalesced array, so that progressive merger should ultimately produce the minimum strain energy configuration. Accordingly, the failure to merge completely indicates an opposing tendency in the convex regions of the kinks, the probable nature of which is indicated in the sketches of Fig 8. From these it is evident that if a volume of crystal containing two arrays of like sign is subjected to tensile stress, then minimum strain energy obtains when the arrays have moved as far apart as possible; hence we conclude that the convex regions are under slight tensile stress. High tensile stress should produce twinning, which was never observed.

The formation of kinks at the $L/4$ point of the rod length has already been shown to be a consequence of the points of inflection of the initial cosine flexure of the rods, for the case of rigidly fixed ends. The $L/3$ location can result from the slightly different case

where one end is rigidly fixed while the other end is merely constrained to axial alignment but remains free to rotate, since in this case the flexure shape⁵ has a point of inflexion at $L/3$. As the single crystal nature of our specimens precluded the possibility of forcing the specimen ends into the aligning holes so as to secure really rigid mounting, it is quite possible that the slight rotations needed to permit the second type of buckling occurred frequently. When both ends of the rod are free to rotate, the flexure shape corresponds to $1/2$ period of a sine function and has no points of inflexion; hence, kinking should not occur, as was confirmed experimentally.

The grip-end kink locations require a different explanation. We shall consider, for the moment, the initial stage of kink generation shown in Fig 7a when a single pair of planar arrays of dislocations of opposite signs have formed at either the $L/4$ or $L/3$ locations and have moved apart the distance s . This movement has displaced the axis in the matrix just above the upper array from the axis just below the lower array, and since the rod ends are still axially aligned, these matrix segments must be resisting bending moments tending to curve them in opposite directions. Minimum strain energy requires that the respective radii of curvature be equal. Let us now assume that the pair of arrays may move as a unit along the rod keeping their separation s unchanged, and inquire whether there exists any more favorable location of lower strain energy. It can be shown that the strain energy is a maximum for the paired array at $L/2$ and that the energy decreases continuously as the paired array moves toward $L = 0$ or 1 , so that, if such migration be possible, the kinks would always be expected to have migrated to the grip-ends long before they attained even microscopically observable size or shape. We conclude, therefore, that the kinks observed at $L/4$ and $L/3$ positions have been prevented from migrating from their points of origin by the surface defects observed, almost without exception, at both kink boundaries in such specimens, while the end-locations occurred only in specimens so nearly perfect that migration of the arrays through the crystal lattice is unimpeded.

In the defect-containing specimens, the kink lengths are certainly determined when the migration of the first

pair of arrays is stopped at the nearest defects. In the other specimens where the movement of the planes of dislocations is apparently unhindered, the formula $s(1 - \cos \alpha)$ suggests that the first pair of planes should separate without limit, to the full length of the specimen. However, we have already seen that the separation of the first pair of arrays creates a bending moment in the rest of the wire, and examination shows that the curvatures in these elastically bent sections produce strain gradients across the glide planes of such nature that they must oppose the advance of the separating pair of arrays. For example, if the dislocations of the upper array in Fig 7a have their extra plane of atoms on the left side of the glide planes, then in the neighborhood of each dislocation the atoms at the left side of the glide plane are compressed because of the extra plane of atoms, and similarly the atoms at the right are under tensile strain. But the rod section ahead of the advancing array is elastically bent with radius of curvature to the left, so that the atoms to the left of the glide planes are already compressed with respect to those at the right, and hence would resist the introduction of dislocations of similar strain gradient. At sufficiently great curvatures the opposing force may reach such magnitude that it becomes easier to generate a new pair of arrays within the undistorted parallelogram than for the first pair of arrays to separate further. Then the first pair would stop separating and fix the ultimate kink length. Under such conditions the kink length would become a function of the specimen length, as our experiments have suggested, for presumably the critical buckling moment would correspond to a definite radius of curvature.

The above theory accounts for the observed locations of the kink bands and for their lengths. It accounts for the fact that in simple kink bands the arcs formed by the bent glide planes always occur in symmetrical pairs, with the arcs at the two convex sides of a band being equal in length; that the grooves always occur in symmetrical pairs with bend angles equal to each other; that the bend angles at the grooves depend upon the lengths of the arcs; that the glide planes at band boundaries may be bent through progressively greater angles the nearer they lie to the grooves. In simple kink bands in zinc these features are all observed.

The planes of dislocations of each successive pair are parallel. The material between them is rotated from its original orientation but remains unbent (or nearly so). On the longitudinal cross-section, with slight or intermediate amounts of compressive strain, there will be a parallelogram within which there is negligible bending. Two diagonally opposite corners of the parallelogram will be located at the grooves at the surface (Fig 4) and the upper and lower sides of the parallelogram will be parallel because there are equal numbers of positive and negative dislocations on the two sides; hence they will be at equal angles to the glide planes between them. With increasing strain, the parallelogram rotates and narrows, becoming finally a line connecting the two grooves, after which no unbent material remains. At all places where glide has occurred it is possible, of course, that faults in stacking of the glide planes are left.

When a single kink band forms, it creates a bending moment on the rest of the rod. There is a tendency for this bending moment to be relieved by the generation of one or more kinks of reversed tilt elsewhere in the wire. When pairs of reverse kinks form, the axes of bending of the glide planes are initially always the same crystallographic direction and are parallel in the two; this has been observed in a rod with as many as four pairs.

To account for secondary boundaries in kink bands, one assumes that successive planar arrays of dislocations may be forced to contact and coalesce over part of their areas at places other than the grooves. Secondary boundaries would be expected when stress conditions change suddenly during the straining of a specimen, for example, when a new kink band starts to form and introduces a bending moment not previously present. Some secondary boundaries may result, also, from planes of dislocations that are stopped at places other than the grooves. This, too, may be a result of local imperfection or changed stress conditions that give a plane of dislocations power to penetrate otherwise insurmountable barriers, or on the other hand, that stop its migration prematurely. Change in rotation axis due to the onset of duplex slip could also be a factor.

Ridges are understandable as the result of planes of like dislocations being forced together, as sketched in Fig 9. Suppose double pairs of dislocations are generated, approximately

simultaneously, with the positive dislocations collecting in planes that are closer to each other than the negative dislocations. As progressive straining forces the positive planes together they come to rest at a plane boundary that extends out to a ridge on the surface. The negative dislocations, meanwhile, form the opposite boundary of the segment. Thus, a ridge is located on the boundary between two crystal segments within which the lattice has rotated in opposite directions about an axis lying both in the plane of the boundary and in the glide plane. During the initial stages of ridge formation while only a single glide system operates, the rotation axis is crystallographically identical to that operating during simple kinking; therefore, the ridges are parallel to the kink grooves. When duplex slip conditions are attained, the rotation axis is altered and the ridge directions become irrational. There is no requirement here that ridges occur in pairs on opposite sides of a kink band, as grooves do, and this is in accord with observation. But there is the requirement that associated with a positive boundary at a ridge there be a negative boundary on each side of a rotated segment, as is, indeed, observed.

It is perhaps surprising that elasticity theory based on the assumption of isotropic medium should hold so well for the present anisotropic crystals. It appears that the chief effect of the anisotropy is to reduce the slenderness ratio at which buckling becomes the mode of failure, for kinking was found to occur even at a slenderness ratio of 1:1. This fact suggests that deformation bands in general may be the result of buckling, for a slenderness ratio of 1:1 would correspond to equiaxed grains.

Summary

1. The formation of kink bands during the compression of single crystal rods of zinc occurs by a progressive rotation of the lattice within the band as deformation proceeds, rather than by a twin-like abrupt shear to a final orientation. Rotations from a few degrees to over 80° have been measured.

2. The width of a simple kink band is fixed by the grooves that form initially on the surface; the bands do not grow wider with increasing deformation as deformation twins do in zinc.

3. These characteristics indicate that kink bands are properly considered as

deformation bands resulting from the ordinary crystal slip process, not as a new mechanism of deformation.

4. In a simple kink the angle of bend of the glide planes at a boundary changes gradually from a maximum at the concave side to a minimum on the convex side. Each boundary is initially a surface containing the direction in the glide plane that is normal to the active glide direction. After severe bending deviations can occur.

5. Complex behavior is often noted: (1) secondary boundaries may radiate out at various angles from points on the original boundaries, and (2) sharp ridges may form in areas subjected to compression. The ridges lie on the boundary between two segments that have rotated in different directions. These complexities are not incompatible with the proposal that kink

bands are special cases of deformation bands.

6. Kink band formation is accounted for by assuming that pairs of dislocations are generated between the boundaries by buckling flexures, the positive dislocations aligning themselves in a plane and the negatives in another plane (a configuration of minimum strain energy). These planes then move apart until stopped by defects or by opposing stresses in the adjoining segments of the rod. Successive planes of dislocations follow similar paths and build up continuously increasing angles of bend.

7. Ridges are accounted for by double pairs of dislocations, with positive dislocations coming together in a plane that extends out to the ridge on the surface, and the corresponding negative dislocations migrating to the

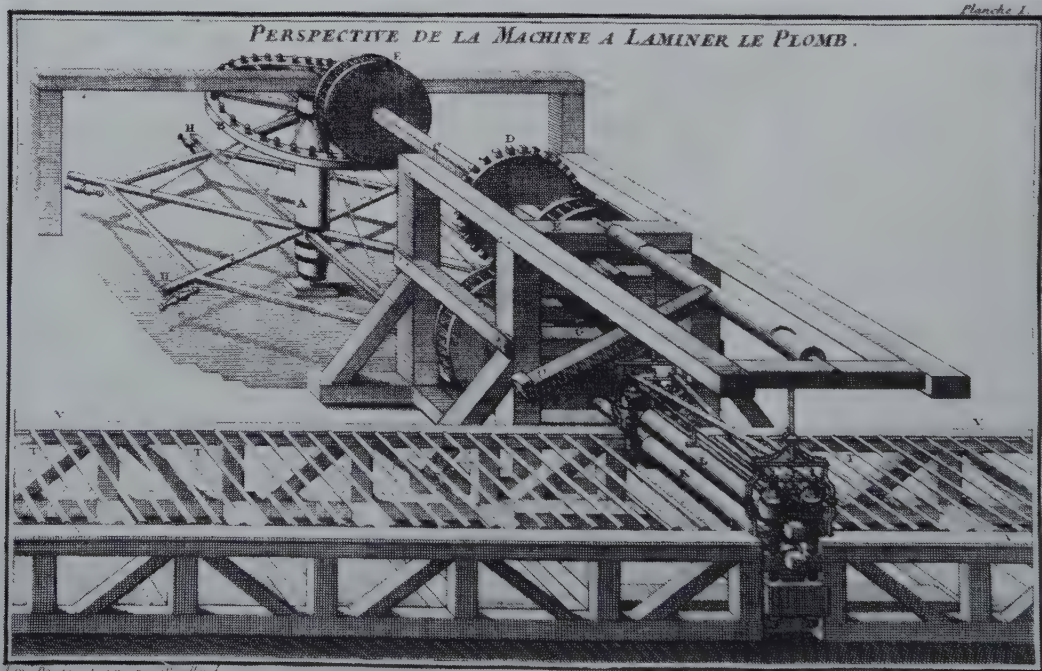
opposite boundaries of two oppositely rotated crystal segments.

Acknowledgment

The authors gratefully acknowledge the assistance of Dr. Werner Bergmann of this laboratory in preparing the long single crystals of predetermined orientation.

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Horse-driven reversing rolling mill for lead roofing sheets.
French, 1760. (Courtesy of C. S. Smith)

Analysis of Interstitial Diffusion Using Activity Methods

A. G. GUY

Thermodynamic activity rather than chemical composition is basic to the analysis of diffusion. This is the essential conclusion reached by Darken¹⁻³ and by Birchenall and Mehl.⁴ If so, it is reasonable to expect that diffusion calculations should be carried out using activities. This paper will illustrate a method of employing activities in analyzing interstitial diffusion.

For interstitial diffusion (of carbon in iron and probably generally) the diffusion of the solvent atoms, volume changes, and other such complexities not explicitly provided for in the usual diffusion equations cause a negligible error of the order of 1 pct. Therefore it is relatively easy to test the validity of an analysis based on activities in the instance of interstitial diffusion. The mathematical treatment of the more complex substitutional diffusion could be simplified once the adequacy of the activity method had been established for substitutional diffusion. Thus the present work has the purpose not only of providing a superior analysis of interstitial diffusion but also of indicating a possible approach to the more important question of an adequate treatment of substitutional diffusion.

Development of the Diffusion Equation

Although activities are convenient quantities to determine experimentally, they are derived from the more fundamental fugacities.⁹ Possible confusion in using activities can be avoided by basing equations involving their use on the relationship,

$$dP = -D_f \frac{\partial f}{\partial x} dt \quad [1]$$

where, dP = the number of grams of

solute crossing one cm^2 in the time dt sec.

D_f = the diffusion constant for use with fugacities; it is assumed to be constant at a given temperature, and has units of secs.

f = the fugacity of the solute in the solid solution; the units are those of pressure, dynes per cm^2 .

x = the distance in cm.

Eq 1 can be considered to be the basic form of the first Fick Law for one-dimensional diffusion.

In order to convert Eq 1 into an equation involving activities, a choice of a standard or reference fugacity, f° , must be made; then,

$$a = \frac{f}{f^\circ} \quad [2]$$

where a is the activity corresponding to the fugacity f . However, the value of a is also determined by the relation,

$$\alpha = \frac{a}{c} \quad [3]$$

where α is the activity coefficient and c is the concentration. For a given value of α (such as $\alpha = 1$) it is evident that the units used for expressing concentration will affect the numerical value of a corresponding to a given amount of carbon dissolved in γ -iron. Since weight per cent concentration is so widely used, this unit will be adopted as the standard in this paper.

When the value of f given by Eq 2 is substituted in Eq 1, the first Fick Law becomes,

$$dP = -D_f f^\circ \frac{\partial a}{\partial x} dt \quad [4]$$

Since $D_f f^\circ$ is constant (at a given temperature), the second form of Fick's Law can be obtained in the usual manner,

$$\frac{\partial c'}{\partial t} = D_f f^\circ \frac{\partial^2 a}{\partial x^2} \quad [5]$$

where c' is the concentration in g per cm^3 . In terms of weight per cent concentration, c , Eq 5 becomes,

$$\frac{\partial c}{\partial t} = \frac{100}{\rho} D_f f^\circ \frac{\partial^2 a}{\partial x^2} \quad [6]$$

where ρ is the density of the solid solution and will be assumed to be constant. In order to simplify Eq 6, it is necessary to consider the standard state, f° .

Standard states are chosen for convenience. There appears to be one especially convenient choice for the analysis of interstitial diffusion. If the activity coefficient, α , is set equal to unity at infinite dilution of the solute,

then $\frac{100}{\rho} D_f f_1^0$ is approximately equal to the usual diffusion constant in dilute solutions. This is true since the activity, a , in Eq 6 can be replaced by the concentration, c , with little error in an infinitely dilute solution.*

Here f_1^0 is the standard fugacity necessary to achieve this standard state. It is proposed that a diffusion constant D_A^1 be defined,

$$D_A^1 = \frac{100}{\rho} D_f f_1^0 \quad [7]$$

where A is the metal whose diffusion is

* The second derivative of a is equal to the second derivative of c in dilute solution only if $\frac{da}{dc}$ is zero. Although theory does not require this condition, in the systems C in Fe, and Zn in Cu, $\frac{da}{dc}$ is in fact found to be essentially zero.

Cleveland Meeting, October 1949.
TP 2604 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received December 31, 1948; revision received June 6, 1949.

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¹ References are at the end of the paper.

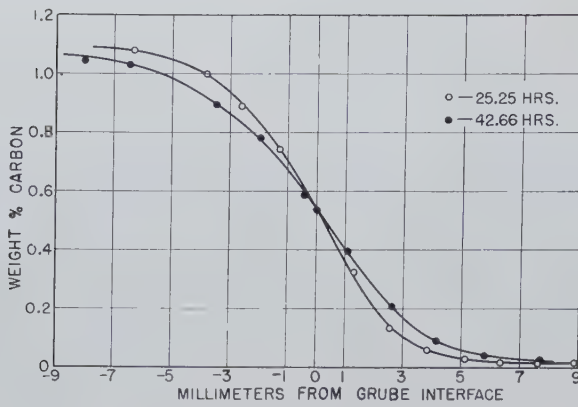


FIG 1—Concentration-penetration curves plotted from the data of Wells and Mehl for the diffusion of carbon in iron at about 1000°C.

being considered. For carbon diffusing in iron this diffusion constant is D_c^1 .

Using the definition of α given above, Eq 6 can be written,

$$\frac{\partial c}{\partial t} = D_A^1 \frac{\partial^2 a}{\partial x^2} \quad [8]$$

This equation would be in a more convenient form for calculation if it were written in terms of a alone, rather than in terms of a and c . From Eq 3,

$$\frac{\partial c}{\partial t} = \frac{\left(1 - \frac{a}{\alpha} \frac{d\alpha}{da}\right)}{\alpha} \frac{\partial a}{\partial t} \quad [9]$$

Therefore, Eq 8 becomes,

$$\frac{\partial a}{\partial t} = Z D_A^1 \frac{\partial^2 a}{\partial x^2} = D \frac{\partial^2 a}{\partial x^2} \quad [10]$$

where Z represents the quantity

$$Z = \frac{\alpha}{1 - \frac{a}{\alpha} \frac{d\alpha}{da}} \quad [11]$$

Since Z is a function of a (and therefore of concentration), D in Eq 10 is not a constant, and the exact solution of this equation involves the use of step-by-step methods.

The Error-function Solution

Fortunately there is an approximate method of applying Eq 10 to determine the value of D_c^1 corresponding to an experimental diffusion curve. The result is found to agree closely with that given by the exact solution—well within experimental error. If the variation of Z is neglected, and Z' is taken as the value corresponding to the median of the range of activities in question, then Eq 10 can be solved in a convenient manner using the error-function solution. When the boundary

conditions are those corresponding to a semi-infinite solid, Eq 10 can be shown to have the solution,⁵

$$a - a_0 = \frac{(a_1 - a_0)}{2} \left[1 \pm \operatorname{erf} \left(\frac{x}{2\sqrt{D't}} \right) \right] \quad [12]$$

where a = the activity corresponding to the carbon content at a distance x from the interface.

a_0 = the activity corresponding to the initial carbon content on one side of the interface.

a_1 = the activity corresponding to the initial carbon content on the other side of the interface.

erf = the Gauss error-function, values of which are given in numerous tables.⁶

$$D' = Z' D_c^1$$

Eq 12 can be used to analyze experimental diffusion data such as those obtained by Wells and Mehl⁷ to determine the value of D' . Since Z' is known, a value for D_c^1 can be obtained using the relation,

$$D_c^1 = \frac{D'}{Z'} \quad [13]$$

The data obtained by Wells and Mehl⁷ for the diffusion of carbon in iron were analyzed using Eq 12 to determine D' and D_c^1 at a number of temperatures. The activities of carbon in austenite (defined by Eq 3 and the choice of standard state) needed for this analysis were calculated from the activity data of R. P. Smith.⁸ Suitable interpolation and extrapolation from his data at 800° and 1000°C were required to cover the temperature range 800°–1100°C in the manner shown in Table 1.

Fig 1 is a conventional plot in terms of weight per cent carbon and distance from the Grube interface* of tests Nos. 9 and 10 of Wells and Mehl. These data can also be plotted in an analogous fashion in terms of activities. Using Eq 12, a value of D' can be calculated for each experimental point of such an activity-penetration plot. Results obtained for points near the ends of the plot are unreliable because of the effect of a small error in activity in greatly altering D' . The values of D' obtained from the five central points of the 42.66 hr data are listed in Table 2 together with the average.

To convert this average value of D' (which is that corresponding to the median activity, 0.642) to the activity diffusion constant D_c^1 , Eq 13 was used. In this instance Z' has the value,

$$Z' = \frac{\alpha}{1 - \frac{a}{\alpha} \frac{d\alpha}{da}} = \frac{1.037}{1 - (0.62)(0.262)} = 1.24 \quad [14]$$

and D_c^1 was determined to be,

*The Grube interface is defined as the plane in the specimen at which the carbon concentration is $\frac{1}{2}(c_1 + c_2)$; that is, where the carbon concentration has its median value. The activity interface is defined in terms of $\frac{1}{2}(a_1 + a_2)$.

Table 1 . . . Carbon Activities in Austenite for a Number of Temperatures

Weight Per Cent Carbon	Temperature, °C			
	800	927	1000	1197
0		0	0	0
0.1		0.102	0.102	0.103
0.2		0.200	0.199	0.200
0.3	0.294	0.298	0.295	0.297
0.4	0.403	0.404	0.400	0.402
0.5	0.517	0.510	0.506	0.513
0.6	0.640	0.620	0.616	0.624
0.7	0.765	0.747	0.742	0.748
0.8	0.902	0.875	0.869	0.877
0.9	(0.88)1.015	1.015	1.007	1.015
1.0		1.163	1.153	1.163
1.1		1.317	1.310	1.323
1.2		1.487	1.476	
1.3		(1.27)1.610	1.652	
1.4			1.842	
1.5			2.050	(1.8)2.70

$$D_c^1 = \frac{3.31 \times 10^{-7}}{1.24} = 2.67 \times 10^{-7} \text{ cm}^2 \text{ per sec [15]}$$

at 1001°C.

Values of D' can be used to calculate approximate penetration curves by means of Eq 12. Fig 2 shows the agreement between the data of Wells and Mehl's test No. 9 (25.25 hr at 997°C) and the penetration curve calculated using the D'^* of Table 2. Somewhat poorer agreement was found in using an analogous approximate method to calculate a carburizing curve in which the concentration range exceeded 1.3 pct C.¹⁰ Exact calculations could be made using the determined value of D_c^1 and Eq 10. However, a step-by-step method of solution would then be employed.

Calculations of D_c^1 at a number of temperatures were made using suitable sets of data obtained by Wells and Mehl. The results are summarized in Table 3. Since it is to be expected that the D_c^1 values will be approximately equal to the usual diffusion constant determined at low carbon contents, the latter values reported by Wells and Mehl for 0.1 pct C are included in the table.

A plot of D_c^1 vs. $\frac{1}{T}$ is shown in Fig 3. The 1102°C point does not fall on the straight line connecting the above three points, possibly because of error in extrapolating the activity data. Assuming that the variation of the diffusion constant with temperature can be written,⁵

$$D_c^1 = Ae^{-\frac{Q}{RT}} \tag{16}$$

the straight line of Fig 3 can be analyzed to give the results,

$$A = 0.17 \text{ cm}^2 \text{ per sec} \tag{17}$$

$$Q = 33,800 \text{ cal per mol} \tag{18}$$

This Q value is practically the same as that found by Wells and Mehl for 0 pct C, 33,300.

Discussion of Results

The advantage of the use of activities in diffusion analysis is that a diffusion constant independent of the concentration of the diffusing element is obtained. Not only is this fact useful mathematically, but it is also of great potential value in the fundamental understanding of metal behavior involving diffusion. However, it is neces-

* The D' value was corrected for the 4°C difference in temperature of the two tests.

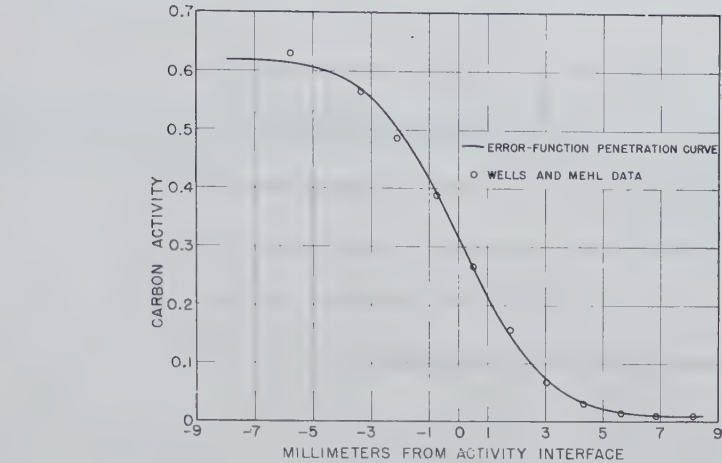


FIG 2—Comparison of the experimental data with the calculated activity-penetration curve for carbon diffusing in iron at 997°C for 25.25 hr.

sary to examine more closely the significance of the multiplicity of permissible activity diffusion constants.

At a given temperature, T , where D_f is constant, Eq 7 shows that the ratio of the activity diffusion constants referred to the two different standard fugacities f_1° and f_2° , is,

$$\frac{D_{f1}'}{D_{f2}'} = \frac{f_1^\circ}{f_2^\circ} \tag{19}$$

But since for a given fugacity,

$$f = a_1 f_1^\circ = a_2 f_2^\circ \tag{20}$$

the ratio is also equal to a_2/a_1 . Similarly, at a different temperature, T' ,

$$\frac{D_{f1}''}{D_{f2}''} = \frac{f_1^{\circ'}}{f_2^{\circ'}} = \frac{a_2'}{a_1'} \tag{21}$$

However, since the ratio a_2/a_1 , will not in general be equal to a_2'/a_1' , it follows that the various activity diffusion constants will not vary in the same ratio with temperature; that is, the activation energies for the various activity diffusion constants will be different.

At first thought this circumstance appears to destroy the usefulness of activity diffusion constants, since the activation energy is a quantity of considerable fundamental significance. Further consideration shows the situation to be quite otherwise. Certainly the activation energy of metal A diffusing in a solid solution would be expected to depend on the concentration of A atoms in the solid solution. Specifically, different values would be expected in a dilute solution (almost pure metal B) than in a concentrated solution (almost pure metal A). It appears reasonable that the Q value determined in a

Table 2 . . . Values of the Activity Diffusion Constant, D' , Determined from the 42.66 Hr Test at 1001°C

Weight Per Cent Carbon	Carbon Activity	Distance from Interface, mm	D' cm ² per sec $\times 10^7$
0.895	1.004	-2.78	3.89
0.783	0.840	-1.26	2.91
0.587	0.602	+0.27	2.83
0.397	0.394	+1.80	3.71
0.208	0.205	+3.33	3.22
		Average	3.31

Table 3 . . . D_c^1 Data for a Number of Temperatures

Temp. °C	Wells and Mehl Test Number	D' $\times 10^7$	Z'	$D_c^1 \times 10^7$	Usual Diffusion Constant Value for 0.1 pct C ⁷ $\times 10^7$
800	13	0.274	1.23	0.222	
925	19	1.41	1.21	1.17	1.1
1001	10	3.31	1.24	2.67	2.6
1102	24	11.1	1.17	9.50	7.7

given instance is that characteristic of the standard state being used. For example, the Q obtained by plotting D_c^1 data applies to the diffusion of carbon in infinitely dilute solution in γ -iron.

Since diffusion constant values applicable for different standard states are in inverse ratio to the corresponding activities, Eq 19 and 20, it is an easy matter to determine the diffusion constant, D^i , characteristic of the standard state in which $\alpha_i = 1$ when the concentration is c_i . Considering diffusion in a solution of concentration c_i ,

$$\frac{D^1}{D^i} = \frac{\alpha_i c_i}{\alpha[c_i]c_i} = \frac{1}{\alpha[c_i]} \tag{22}$$

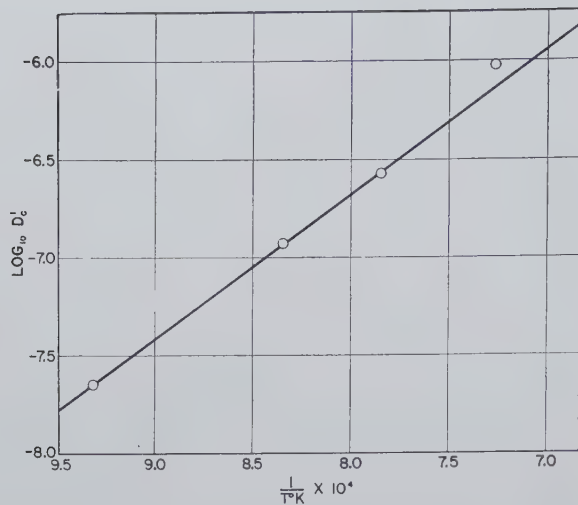


FIG 3—The variation of D_c^1 with temperature.

where $\alpha[c_i]$ represents the value of the activity coefficient for use with dilute solutions evaluated at c_i . Using Eq 22, activation energies of 33,700 and 33,300 were calculated for 0.4 and 0.8 pct C standard states by means of the D_c^1 values at 800° and 1001°C of Table 3 and the activity data of Table 1. These are in qualitative agreement with the results obtained by Wells and Mehl. The Q value of 42,000 given by Birchenall and Mehl⁴ is of questionable theoretical significance since it was obtained using a different standard fugacity for each temperature.

A matter that has caused some concern in previous attempts to analyze the diffusion of carbon in iron has been the "interface anomaly." This term describes the fact that the respective interfaces on which the Grube and Matano solutions are based fail generally to coincide. The question then arises as to which of these is the significant interface.

There seems little doubt that the initial interface (in the Kirkendall sense) is the significant one. The correspondence of other interfaces with this one can be summarized as follows, assuming negligible density change:

1. The Grube interface should coincide with the initial interface in perfect interstitial solid solutions. The solution of carbon in iron is not perfect, and an appreciable difference between the two interfaces is found.

2. As a consequence of its definition, the Matano interface* must coincide with the initial interface during the

course of diffusion in interstitial solid solutions.

3. The interface defined by the median activity (the Grube interface marks the median concentration) will coincide with the original interface when Z (Eq 10) is constant. In the Fe-C system the variation of Z is sufficient to cause absence of perfect coincidence.

The relation between the usual diffusion constant, D^* , and the activity diffusion constant, D_A^1 , is easily derived by equating Eq 8 and the usual form of the second Fick Law;

$$\frac{\partial c}{\partial t} = D_A^1 \frac{\partial^2 a}{\partial x^2} = \frac{\partial}{\partial x} \left(D^* \frac{\partial c}{\partial x} \right) \quad [24]$$

Integrating the second two terms gives,

$$D_A^1 \frac{\partial a}{\partial x} = D^* \frac{\partial c}{\partial x} \quad [25]$$

since the constant of integration must be zero. By analogy with Eq 9, Eq 25 can be written,

$$D_A^1 Z \frac{\partial c}{\partial x} = D^* \frac{\partial c}{\partial x} \quad [26]$$

or

$$D^* = Z D_A^1 \quad [27]$$

This equation is merely another form of the relation given by Birchenall and Mehl.⁴

Summary

1. The use of the fugacity concept has permitted a more satisfactory treatment of interstitial diffusion by activity methods.

2. Of the multiplicity of possible ac-

tivity diffusion constants, it has been proposed that D_A^1 , corresponding approximately to the usual diffusion constant in dilute solution, be adopted as standard for analysis of interstitial diffusion.

3. An equation that permits the direct treatment of activities has been developed. A convenient approximate solution of this equation was used to analyze the data of Wells and Mehl to determine diffusion constant values. The results agreed well with those obtained by Wells and Mehl.

4. It was suggested that there is an activation energy associated with each choice of standard fugacity. That found for D_c^1 (standard fugacity being that of an infinitely dilute solution) was 33,800, in good agreement with Wells and Mehl's extrapolated value for 0 pct C.

5. Further support for the view that the activity rather than the concentration is the basic factor in diffusion has been provided. Specifically, activity diffusion constants independent of concentration have been developed for possible application to the analysis of the more complex substitutional diffusion.

Acknowledgments

The author thanks Dr. R. F. Mehl for suggesting the problem that eventually led to this study of activity methods; Dr. K. O. Beatty, Jr., for indispensable aid with the fugacity concept; Mr. J. L. Sox for carrying out many of the calculations; and Dr. W. G. Van Note, Director of the Engineering Research Department, North Carolina State College, for his counsel and aid.

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* For interstitial diffusion the Matano interface definition is such that the carbon atoms gained by one side equal those lost by the other.

The Transformations in β -CuAl Alloys*

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The transformations in eutectoidal systems have been extensively studied as they occur in steels.¹ As a consequence of these studies the martensite, bainite and pearlite reactions found for most steels are widely recognized. Further, because of the work of Smith and Lindlie² and of Greninger and Troiano³ these reactions, except for the bainite reaction, are considered as typical for all eutectoidal systems.

The primary alloy studied by Smith and Lindlie² was a CuAl alloy of near eutectoid composition as was the alloy more recently studied by Mack.⁴ The transformations in these alloys were, for the most part, studied metallographically, while an X ray analysis of structures was essentially wanting. It has appeared desirable, therefore, to investigate selected CuAl alloys at and slightly away from eutectoid composition. For this purpose three alloys were prepared containing 10.5, 11.9 and 13.5 pct Al respectively. The results of a metallographic and X ray analysis of structures obtained for isothermal transformation of the β -phase are reported here for the 11.9 and 13.5 pct Al alloys.

Introduction

The phase equilibria in the CuAl alloys of immediate concern are indicated in the equilibrium diagram presented in Fig 1. § The equilibrium and transition structures which have been reported in this system are as follows:

- α = copper rich terminal phase (f.c.c.)
- β = high temperature eutectoidal phase (b.c.c.)
- γ_2 = aluminum-rich intermediate phase (γ -brass structure)
- α' = modified α -phase postulated by Bollenrath and Bungardt.²⁶

§ Taken from Metals Handbook (1948).

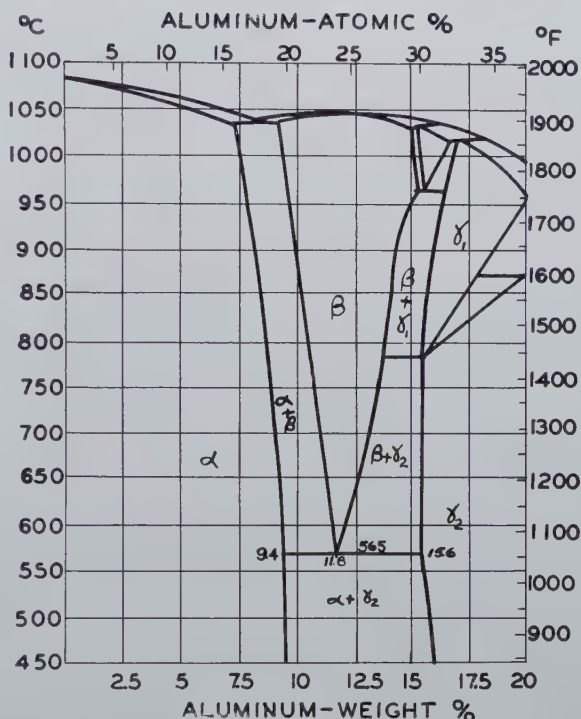


FIG 1—Phase diagram for eutectoidal Cu-Al alloys. *Metals Handbook—1948.*

- β_1 = ordered β (b.c.c.)
- β' = martensite structure: Al < 13 pct (near h.c.p.)
- β'' = β' of Smith and Lindlie²
- γ' = martensite structure: Al > 13.5 pct (h.c.p.); β_2 of Bollenrath and Bungardt.²⁶

Cleveland Meeting, October 1949.
TP 2573 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received December 2, 1948; revision received May 23, 1949.

* Most of the material presented in this paper has been taken from a thesis submitted by S. M. Grymko in partial fulfillment of the requirements for the degree of Master of Science, The Pennsylvania State College, January 1949.

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¹ References are at the end of the paper.

The eutectoid transformation can be suppressed at sufficiently high cooling velocities. The β -phase is then retained to about 500°C where ordering sets in. The ordering interval is not satisfactorily known but indirectly appears to be non-suppressible. Subsequent to the ordering reaction and consequently at lower temperatures, the ordered β_1 decomposes to a martensitic structure. The kinetics of these reactions and in particular those of the martensite reaction have an important bearing on the results obtained in this investigation. Since some confusion exists concerning certain reported aspects of these reactions, they will be considered in some detail.

THE MARTENSITE REACTION

The martensitic structure is formed on cooling through a critical range⁶

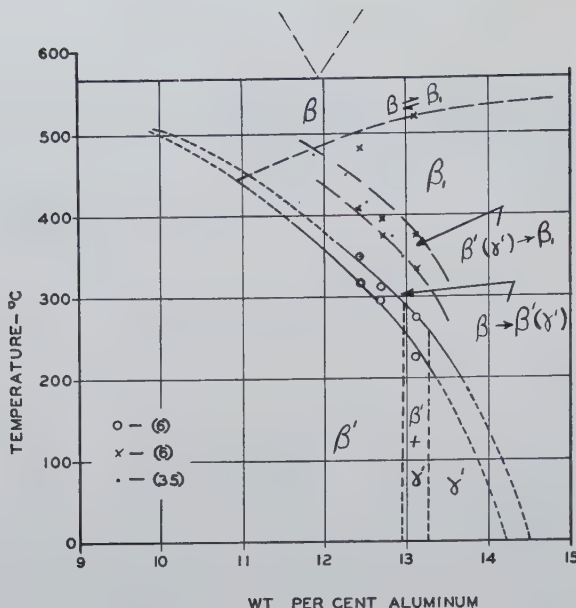


FIG 2—The effect of composition on the sub-critical transformations in eutectoidal Cu-Al alloys.

much as is martensite in steels.^{12,3} The Ar'' temperature for the CuAl alloys depends on the composition and decreases with increasing aluminum content.⁹ In certain composition ranges Ar'' is cooling velocity dependent⁸ although it is pointed out that this dependence on cooling velocity may be indirect. Thus the indicated variation in Ar'' may result from a modification of the ordering reaction with cooling velocity. At a composition of 14.5 pct Al the martensite range is entirely below room temperature but lies above -190°C .¹⁰ However, due to the reversibility of this reaction ($\beta_1 \rightleftharpoons \gamma'$) the martensitic structure is not retained at room temperature.

The inverse martensite reaction, that is, the formation of β_1 from the martensitic structure takes place on suitable heating of the martensitic structure. This reaction has been established by metallographic¹⁴ dilatometric^{7,15} and X ray^{5,6} analyses of the structures at temperature and lies above the martensite range on cooling. These data are summarized in Fig 2. The inverse martensite reaction is probably sensitive to heating velocity, but few data are available for consideration on this point.

THE STRUCTURE OF THE CuAl MARTENSITES

The microstructures of the CuAl martensites have been adequately described by Greninger.¹¹ It is sufficient to point out that two structures β' and γ' are separable on the basis of

metallographic differences. The striations in γ' arise from twin formation while those in β' seemingly do not.

The crystal structures of the martensites are close to hexagonal close packed. The departures from the close packed lattice, observed for the β' structure, are notable at low aluminum contents. Thus while the lattice for γ' containing 13.5 pct Al is described as a hexagonal close packed structure, the lattice for β' is described as differing from γ' in that the $[00.1]$ direction is at an angle of 2° to the (00.1) plane normal, and the angle between the planes (10.0) and (01.0) differs about 1° from 120° .¹³ Because of this departure from the hexagonal close packed structure the X ray pattern for the β' structure is markedly different from that for the γ' structure. This is very important as it affords an unambiguous means of determining qualitatively the compositions of the martensitic structures in partially transformed alloys without requiring the precision measurement of lattice constants.

THE REACTION $\beta \rightarrow \beta''$

The structure β'' as noted before was discovered by Smith and Lindlie² on the isothermal transformation of β for an eutectoidal alloy. Smith and Lindlie on the basis of limited X ray evidence have considered this structure as being aluminum dilute apparently tending towards the composition of the α -phase.

More recently Mack⁴ has considered this structure as being β_1 or the

ordered β structure and has defined its range of occurrence in an 11.9 pct Al alloy from about 525 to 93°C . On the basis of the discussion of the kinetics of martensite formation it is evident that the reasoning which led Mack to place the lower limit of the martensite range at 93°C is faulty.

It is emphasized that β'' is not β_1 . A specimen of 11.9 pct Al alloy quenched from 950°C is an ordered structure but does not reveal the metallographic structure β'' .

THE ISOTHERMAL TRANSFORMATION DIAGRAM FOR EUTECTOIDAL CuAl ALLOYS

Isothermal transformation diagrams in comparable eutectoidal alloys have been presented by Smith and Lindlie² and by Mack.⁴ These diagrams are in good agreement except for the martensite range which has been modified by Mack to conform with more recent knowledge about this reaction. Beyond this discrepancy both diagrams describe a pearlite range and a β'' range which stand in good agreement where comparable.

From the discussion of the inverse martensite reaction it appears that certain of Mack's tempering data may require re-evaluation.

The works of Smith and Lindlie and of Mack are the only ones in which the isothermal transformations in eutectoidal CuAl alloys have been reported in detail.

Experimental Procedure

MATERIALS

The alloys for this investigation were prepared from high purity aluminum and OFHC copper. Melting was carried out in a graphite crucible under graphite. The molten alloys were stirred several times and were then chill cast. The ingots were about $\frac{1}{4} \times 2 \times 3$ to 5 in. and were very coarse grained. These ingots were hot-rolled in part to $\frac{1}{8}$ and $\frac{1}{32}$ in. thicknesses.

The ingots were analyzed for copper by both chemical and electrolytic methods. The aluminum content was determined by difference. The results are presented in Table 1.

ISOTHERMAL STUDIES

Sliver specimens ($\frac{1}{32} \times \frac{1}{32} \times 1$ in.; $\frac{1}{8} \times \frac{1}{32} \times 1$ in.) were heated at 950°C for 10 min. in a vertical tube

furnace in an atmosphere of purified nitrogen. The specimens were quenched into a small lead furnace at the desired temperature and after holding were quenched in a 10 pct aqueous solution of sodium hydroxide. The temperature of the quenching bath was controlled to $\pm 1\frac{1}{2}^{\circ}\text{C}$. A lead-tin bath was used for the lowest transformation temperatures.

METALLOGRAPHIC EXAMINATION

The sliver specimens were polished in clamps with the final surface being obtained with magnesium oxide (Shamva) on a selvyt cloth. The specimen was etched in a fresh 1:1 solution of NH_4OH and 3 pct H_2O_2 .

X RAY EXAMINATION

The metallographic specimens were polished and etched down to slivers about $\frac{1}{64}$ in. diam. Debye-Scherrer diagrams using $\text{Cu-K}\alpha$ radiation (Ni filter) were obtained. Standard β' and γ' patterns were obtained on the 10.5, 11.9, and 13.5 pct Al alloys as quenched from 950°C into the aqueous NaOH solution. Suitable α and γ_2 reference patterns were obtained on the isothermally transformed alloys.

REPRESENTATION OF TEST RESULTS

The data have been presented in the form of isothermal transformation diagrams. Suitable micrographs and X ray diffraction patterns have been presented.

Experimental Results

Because of the emphasis with which the alloy of eutectoid composition has been studied, it appeared desirable to devote the major effort in this investigation to a study of the 13.5 pct Al alloy. The transformations in this alloy, therefore, will be considered first.

THE 13.5 PCT AL ALLOY

Metallographic Results

The isothermal transformation diagram for this alloy is presented in Fig 3. There are certain striking features to this diagram. Firstly, the temperature of maximum transformation rate for practical purposes coincides with the eutectoid temperature. Secondly, it has appeared necessary to make use of four transformation

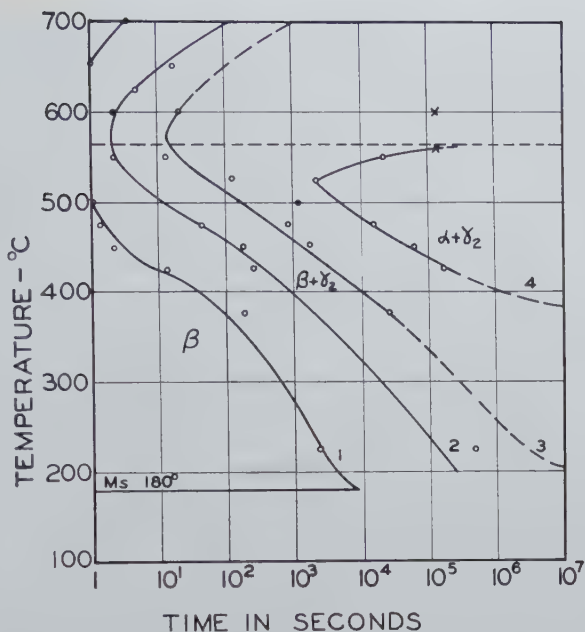


FIG 3—The isothermal transformation diagram for a 13.5 pct Al-Cu alloy.

Table 1 . . . Analyses of the Alloys

Alloy	Pct Al by Electrodeposition	Pct Al by Chem. Analysis
11.9	11.90	11.85
	12.00	11.86
	12.00	12.00
13.5	13.60	13.40
	13.52	13.43
		13.58

lines to describe adequately the course of isothermal transformation, and thirdly, no pearlite reaction is indicated. These facts may best be appreciated from a consideration of the microstructures.

In Fig 4 and 5 are presented the structures corresponding to beginning and end of the first reaction at 550°C as indicated by lines 1 and 2 on the isothermal transformation diagram. The precipitate structure is truly acicular and seemingly forms on the $\langle 100 \rangle_{\beta}$ directions. The structures formed by this reaction are not stable structures and rapidly undergo alteration at this temperature. The alteration seemingly is a species of agglomeration and growth, which, as will be pointed out, is not a simple process. The third line on the diagram indicates the start of agglomeration while the fourth line indicates the time for completion of the precipitation-growth process. Further holding causes no apparent change in this final structure except for a marked coarsening. These final alterations are indicated in Fig 6 to 8. There has been no pearlite structure formed during the course of transformation. This is emphasized by a

consideration of Fig 9 which reveals the nearest approach to a pearlitic structure which has been observed in this alloy on isothermal transformation.

The transformation sequence revealed in the above micrographs is typical except for minor variations for all structures formed below the eutectoid temperature. As is implied by the fourth line on the transformation diagram, transformation above the eutectoid temperature differs in certain important respects.

The structure existing at the end of the first reaction at 650°C is presented in Fig 10. The structure existing at the end of the second reaction is presented in Fig 11. The final structure under polarized light is seen to have a matrix of an acicular nature, Fig 12. This sequence of structures emphasizes the role of the first reaction in leading to transitional structures which in turn lead to the formation of the final equilibrium structures.

The initial and final structures are modified as a function of the transformation temperature but at all temperatures the structures arising from the first reaction are closely related microstructurally.

At the highest temperatures studied the initial reaction did not lead to complete decomposition of the matrix, Fig 13. This is to be expected since presumably above the eutectoid temperature only a proeutectoid constituent should precipitate. The character of the first reaction cannot be

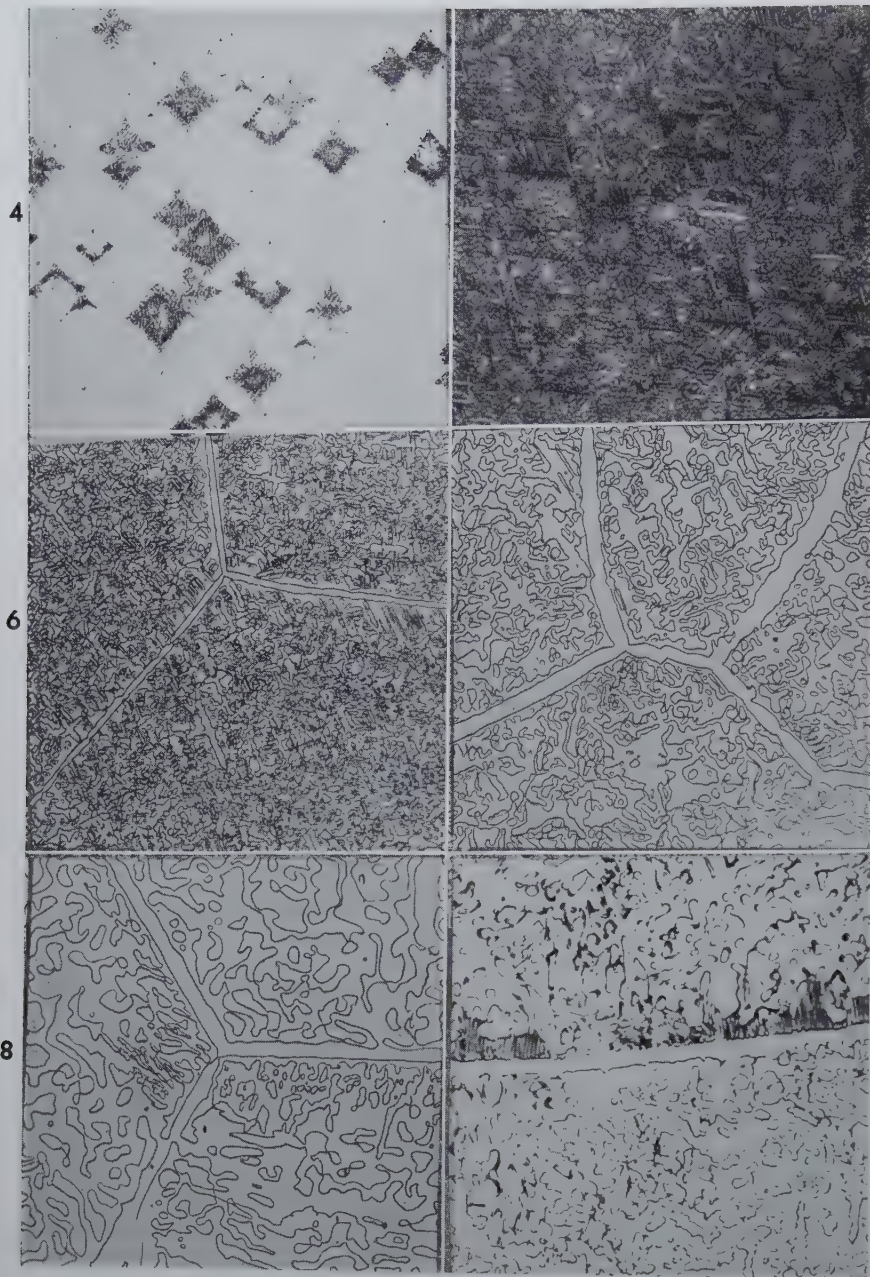


FIG 4—13.5 pct Al-Cu specimen transformed at 550°C for 1 sec. $\times 250$
 FIG 5—13.5 pct Al-Cu specimen transformed at 550°C for 3 sec. $\times 250$
 FIG 6—13.5 pct Al-Cu specimen transformed at 550°C for 32 min. $\times 250$
 FIG 7—13.5 pct Al-Cu specimen transformed at 550°C for 9 hr. $\times 250$
 FIG 8—13.5 pct Al-Cu specimen transformed at 550°C for 1 week. $\times 250$
 FIG 9—13.5 pct Al-Cu specimen transformed at 425°C for 24 hr. $\times 250$

elucidated on the basis of metallographic evidence, however.

As the transformation temperature is decreased the microstructures become finer, Fig 14 to 21. At temperatures increasingly lower than the eutectoid temperature the first reaction becomes increasingly sluggish. The times for the completion of the precipitation-growth reaction below the eutectoid temperature develop a C-shaped curve which is comparable to that developed for the pearlite reaction in steels.¹⁶ The temperature for minimum reaction time

does not coincide with the analogous temperature for the first reaction but lies below the eutectoid temperature. From this it may be concluded that stable end structures arise as a consequence of the second reaction.

These data may be summarized briefly before the X ray data are considered. The metallographic data indicate that two reactions are encountered on isothermal transformation in this alloy, one of which leads to metastable structures as indicated by its occurrence through the eutectoid tem-

perature range. After completion of this reaction a second reaction sets in and leads to the complete modification of the microstructures with the ultimate formation of stable end products. Both reactions are represented by a combination of C-shaped curves and differ in that the eutectoid temperature affects the location of the stable structures completion curve.

X Ray Results

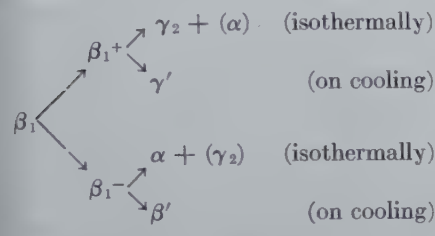
Four different crystal structures, indicated by X ray analysis may be obtained together in the transformed specimens, namely those of α , γ_2 , β' , and γ' . Positive evidence of β_1 was not obtained for the 13.5 pct Al alloy. The diffraction patterns characteristic of these four crystal structures are presented in Fig 22 to 25. The spotted lines in the pattern in Fig 23 arise from the γ_2 lattice while the continuous lines arise from α . Many of the lines in the β' and γ' patterns correspond closely to lines in the α and γ_2 patterns. Strong lines which are characteristic for the respective structures are indicated on the patterns. All diffraction patterns were checked for these strong lines. The presence or absence of the respective lines was taken as indicating the presence or absence of the respective structures.

The pattern for the β' structure was obtained from an 11.9 pct Al specimen quenched from 950°C into water + 10 pct NaOH. From the presence of the weak lines at low θ values it is evident that this structure is ordered. The γ' structure likewise is ordered.

The results of the X ray analysis are summarized in Table 2. Important diffraction patterns of the structures reported in Table 2 are presented in Fig 26 and 27. These data indicate the following sequence of composition adjustment in the transforming specimens. Prior to the start of the first reaction, the structure as quenched is γ' with possible traces of β' , Fig 25. Through the course of the first reaction the structure in the quenched specimens becomes a mixture of γ' and β' , Fig 26. This structure exists at the times corresponding to completion of the first reaction which means that the γ' revealed is not matrix γ' , or γ' which has been unaltered in composition. Rather because of the formation of large amounts of β' it is necessary that the γ' formed be very rich in aluminum. The γ' structure is next replaced by γ_2 and the structure is γ_2 and β' , Fig 27. For the fully transformed alloy the

structure is α and γ_2 , Fig 23.

The first reaction leads to the formation of structures in the as quenched specimens, which, it is known, cannot be formed at the temperature of isothermal holding. Since β' and γ' form from β_1 (or β) of characteristic composition it is concluded that the structures resulting from the first reaction prior to the quenching process consist of β_1 of two compositions, namely, β_1^+ which is aluminum rich and β_1^- which is aluminum depleted. There are thus two β_1 structures, one tending toward the composition of α , the other toward the composition of γ_2 . It is emphasized that these structures have not precipitated at temperature, rather the process throughout is a diffusion process alone. The β_1^+ becomes enriched in aluminum to the point where it can precipitate as γ_2 . The precipitation of γ_2 at the expense of the aluminum content of the β_1^- continues to the point where α can precipitate. The final structure then being α and γ_2 . This sequence of events is indicated as follows:



The reactions on isothermal holding seemingly are not interdependent. It is assumed that the same is true for the reactions on cooling.

The transformation sequence described here is in complete harmony with that for the transformation of austenite to bainite in steels.¹⁶⁻¹⁸ The composition of the structures and the manner in which the necessary composition adjustments are achieved in this CuAl alloy, however, are much more easily determined than is true for the steels.

THE 11.9 PCT Al ALLOY

Metallographic Results

The transformation data for this alloy for stock thicknesses of $\frac{1}{8}$ and $\frac{1}{32}$ in. are presented in the isothermal transformation diagram in Fig 29.

This isothermal transformation diagram, above the martensite range, is in good agreement with those diagrams presented by Smith and Lindlie² and

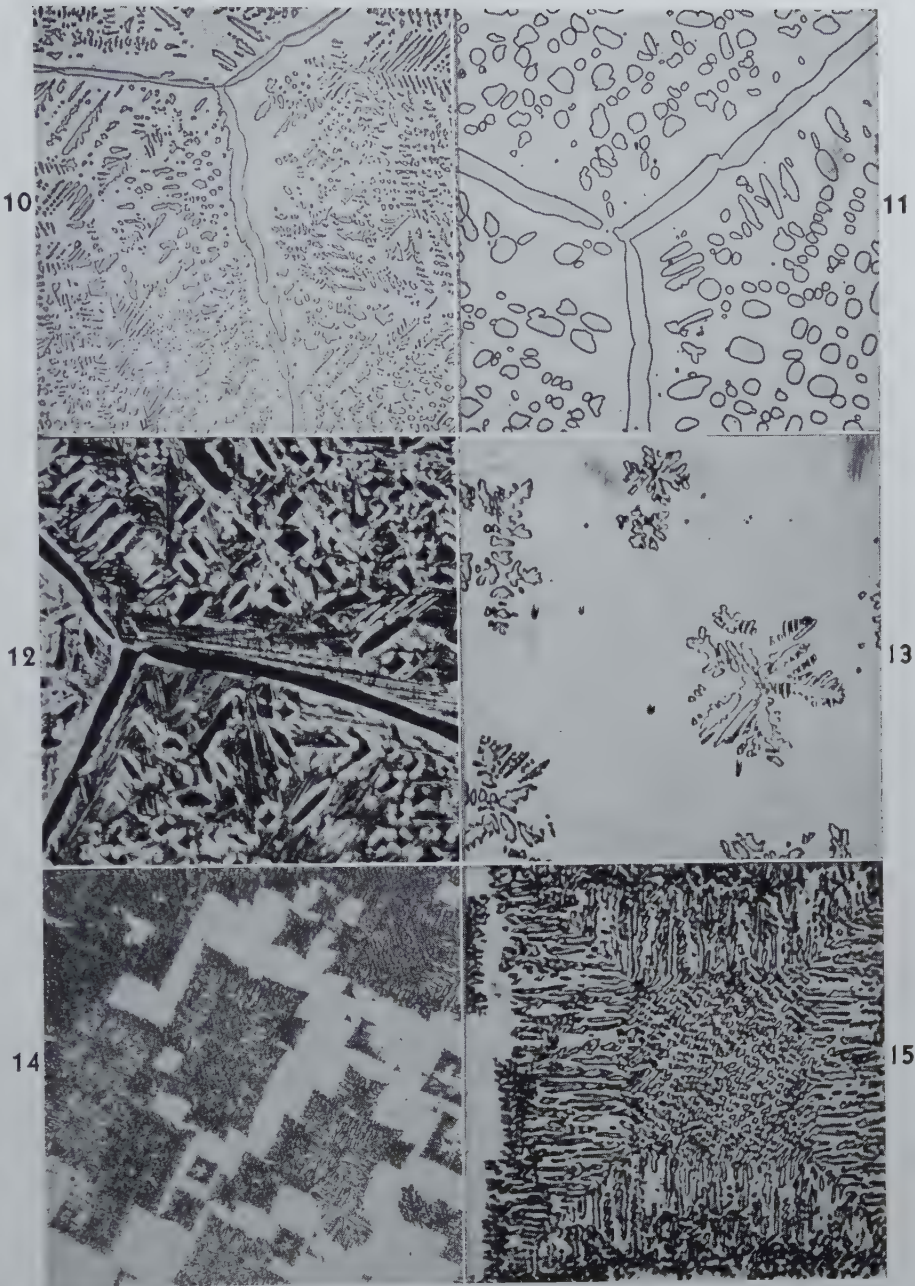


FIG 10—13.5 pct Al-Cu specimen transformed at 650°C for 5 min. $\times 250$
 FIG 11—13.5 pct Al-Cu specimen transformed at 650°C for 2 hr. $\times 250$
 FIG 12—13.5 pct Al-Cu specimen transformed at 650°C for 2 hr. Polarized light. $\times 250$
 FIG 13—13.5 pct Al-Cu specimen transformed at 700°C for 5 sec. $\times 1000$
 FIG 14—13.5 pct Al-Cu specimen transformed at 600°C for 1 sec. $\times 250$
 FIG 15—13.5 pct Al-Cu specimen transformed at 600°C for 1 sec. $\times 1000$

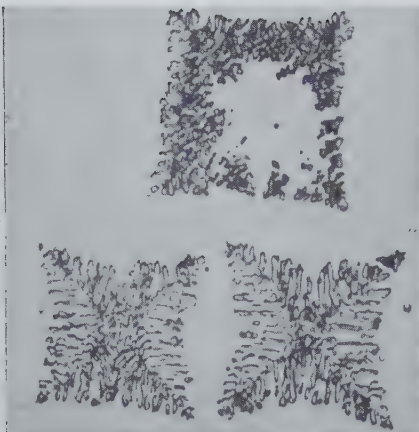
Mack.⁴ The γ_2 precipitate observed by Mack in the high temperature range of transformation is not observed for this alloy. The martensite range is presented after the data of Gawranek et al.⁹

In the intermediate range in which β'' and α are formed it is interesting to note that some disagreement in the times for the β'' and α reactions exists for the two materials. The increased working or possibly the reduced grain size of the $\frac{1}{32}$ in. thick alloy promotes

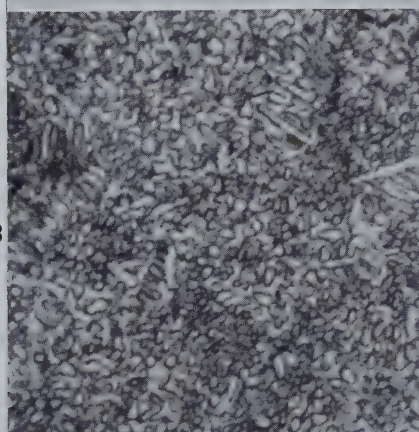
more rapid transformation in the intermediate range. The results for the $\frac{1}{32}$ in. thick stock are in satisfactory agreement with the results of Smith and Lindlie,² and with those of Mack where comparable.

The metallographic data for the pearlite structures are interesting with respect to the pearlite spacing. It is generally accepted on the basis of pearlite spacings measurements on steels¹⁹⁻²³ that the pearlite structures become finer with decreasing tempera-

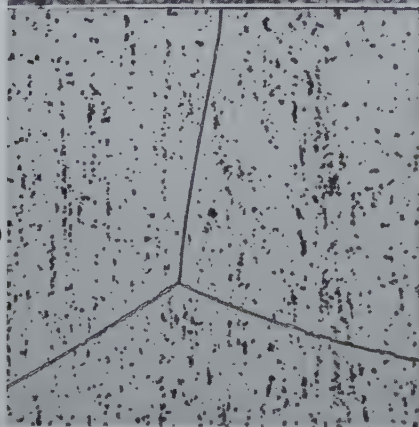
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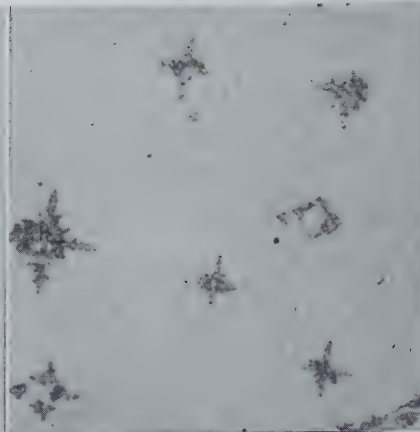
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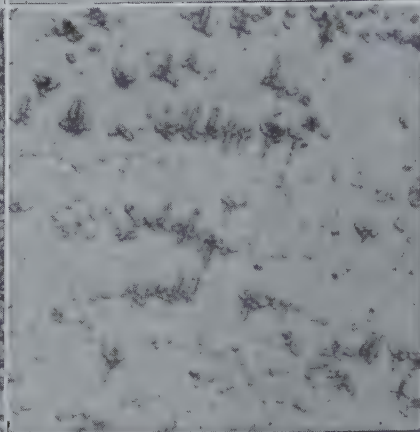
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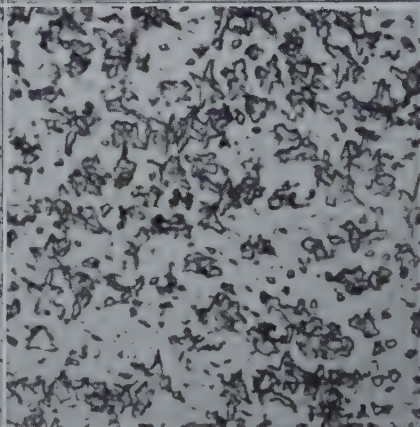


FIG 16—13.5 pct Al-Cu specimen transformed at 550°C for 1 sec. $\times 1000$
 FIG 17—13.5 pct Al-Cu specimen transformed at 475°C for 8 sec. $\times 1000$
 FIG 18—13.5 pct Al-Cu specimen transformed at 425°C for 60 sec. $\times 1000$
 FIG 19—13.5 pct Al-Cu specimen transformed at 375°C for 4 min. $\times 1000$
 FIG 20—13.5 pct Al-Cu specimen transformed at 225°C for 24 hr. $\times 250$
 FIG 21—13.5 pct Al-Cu specimen transformed at 225°C for 1 week. $\times 1000$

ture of formation. This is evidently not the case for the pearlite spacing in the CuAl alloy under investigation. For this alloy the pearlite spacing first decreases with decreasing temperature of formation. After passing through a minimum value it next increases. Thus the functional relationship between pearlite spacing and temperature cannot have the same form for this alloy as it apparently has for steels.^{20,23}

In a consideration of the relationship between pearlite spacing and temperature it is pointed out that for steels the

pearlite structures are divided into the coarse lamellar types and into the rosette (lamellar) or troostitic types. The first named occur well above the nose of the "S" curve while the second occur at about the nose of the "S" curve. (It has been pointed out that pearlitic structures as yet inadequately defined occur below the nose of the "S" curve.^{16,18}) As the temperature for pearlite formation decreases the interlamellar spacing decreases. This change in dimensions of the pearlite has been advanced as indicating a limiting car-

bide nucleus size,^{20,23} and this carbide nucleation has been used to account for the pearlite reaction. By an extension of this argument to conform to the requirements of the CuAl alloy investigated here, it would be expected that microstructural variations in the pearlitic structures would be analogous to those observed in steels. Such is not the case, however. Thus the coarse pearlites are replaced at lower temperatures by nodular pearlites—at about the "nose" of the C-curve—but these in turn give way at still lower temperatures to coarse lamellar pearlites, Fig 30 to 32. Thus the kinetics of pearlite formation in this alloy cannot be accounted for by simple comparison with the analogous reaction in steels.

X Ray Results

X ray data taken on specimens of the 11.9 pct Al alloy transformed at several temperatures are presented in Table 3. The patterns taken for transformation in the intermediate range, that is, the range in which β'' and α are formed indicate that the reaction in this range is essentially the same as that encountered in the 13.5 pct Al alloy for the first reaction. The major difference appears to be the change in the sequence in which the precipitate structures appear on isothermal transformation. Thus the structure existing in specimens quenched before α precipitates is β' . The structure after longer holding is $\alpha + \beta'$ which, on further holding, is replaced by a mixed structure of α , β' , and γ' . The amount of γ' formed is relatively small as is indicated in Fig 28. The final structure is $\alpha + \gamma_2$. The γ_2 lines are very weak.

Discussion of Experimental Results

The pearlite reaction has been considered as a reaction to be expected in general in the course of the subcritical decomposition of a high temperature eutectoidal phase. That this reaction is not generally obtained is indicated by the results presented here in the study of the 13.5 pct Al alloy. Results of a similar character have recently been reported by Hibbard et al²⁴ and by Sheehan et al.²⁵

In the CuAl alloys it appears that the pearlite reaction cannot be obtained at aluminum contents of 10.5 pct* and less or at 13.5 pct and greater. This is

* Further work on this alloy is contemplated. Results to date indicate the absence of the pearlite reaction.

24a

 β'

26b

 $\beta' + \gamma'$

25c

 γ'

22d

 $\alpha + \gamma_2$

28e

 $\alpha + \beta' + \gamma'$

23f

 $\gamma_2 + \alpha$

27g

 $\gamma_2 + \beta'$

Fig 22-28—X ray Diffraction Patterns

FIG 22—(d) Alloy 11.9 pct Al held 1 hour at 500°C
 FIG 23—(f) Alloy 13.5 pct Al held 3 hours at 525°C
 FIG 24—(a) Alloy 11.9 pct Al held 60 seconds at 500°C
 FIG 25—(c) Alloy 13.5 pct Al held 1 hour at 225°C
 FIG 26—(b) Alloy 13.5 pct Al held 48 hours at 225°C
 FIG 27—(g) Alloy 13.5 pct Al held 6 seconds at 500°C
 FIG 28—(e) Alloy 11.9 pct Al held 8 minutes at 450°C

(Note: Standard patterns are indicated by underlines. Characteristic diffraction lines are indicated by vertical lines on the respective standard patterns.)

not readily explainable on the basis of the current concepts of the pearlite reaction for seemingly about 50 pct of the β -phase in both the 10.5 and 13.5 pct Al alloys should be available for pearlite formation.

The preliminary reaction which is obtained in the 13.5 pct Al alloy on isothermal holding seemingly is intimately related to the β'' reaction in the 11.9 pct Al alloy. Thus for both alloys it appears that the diffusion of aluminum takes place prior to any crystal structure change in the matrix. If the alloy is cooled before the crystal structure change in the aluminum rich or depleted zones can take place, the crystal structure change is effected during the quenching cycle. The structure at room temperature then is a martensitic structure, one made up of mar-

tensites of the various compositions of the transforming β_1 . Further since the crystallography of these zones of aluminum enriched and depleted β is fully determined, the martensitic structures formed during the quenching cycle are restricted by this crystallography. Thus neither β' nor γ' have the orientation habits determined by Greninger.¹¹ Since it is possible that the lattice relationships for the structures examined here and those examined by Greninger are the same, there arises the possibility that the martensite transformation mechanism varies with pretreatment of the matrix.

The first reaction in the 13.5 pct Al alloy and the β'' reaction in the 11.9 pct Al alloy are reactions which conform in all major details to the proposed bainite reaction¹⁷ in steels. In the 13.5

pct Al alloy the β_1 structure is modified by diffusion processes such that aluminum enriched and depleted zones are set up. These regions become evident at room temperature under metallographic examination, because of the different etching characteristics of the β' and γ' .

After the β_1^+ and β_1^- structures are formed, both decompose but evidently by more complicated processes than were observed for the bainite reaction in steels. This follows from the fact that the final structure is markedly different from the structure arising as a consequence of the first reaction. Thus if the γ_2 (in the 13.5 pct Al alloy) were formed in the matrix by a shearing mechanism such as is postulated for the bainite reaction in steels, a supersaturated γ_2 structure would be obtained and this

Table 2 . . . Structures by X ray Diffraction in Transformed Specimens of Alloy 13.5 Pct Al

700°C		600°C		550°C		525°C		500°C	
Time	Structure	Time	Structure	Time	Structure	Time	Structure	Time	Structure
80 hr	$\beta' + \gamma_2$	1 sec	$\gamma_2 + \beta'$	1 sec	$\gamma_2 + \beta'$	8 sec	$\gamma_2 + \beta' + (\gamma')$	5 sec	$\beta' + \gamma'$
		3 sec	$\gamma_2 + \beta'$	32 min	$\gamma_2 + \beta'$	16 sec	$\gamma_2 + \beta' + (\gamma')$	15 sec	$\beta' + \gamma'$
		10 sec	$\gamma_2 + \beta'$	2 min	$\alpha + \gamma_2$	2 min	$\alpha + \gamma_2 + (\beta')$	30 sec	$\beta' + \gamma' + (\gamma_2)$
Time	Structure	30 sec	$\gamma_2 + \beta'$	9 hr	$\alpha + \gamma_2$	30 min	$\alpha + \gamma_2$	60 sec	$\beta' + \gamma_2 + (\gamma')$
5 sec	$\beta' + \gamma'$	2 min	$\gamma_2 + \beta'$	1 wk	$\alpha + \gamma_2$	3 hr	$\alpha + \gamma_2$	120 sec	$\beta' + \gamma_2$
8 min	$\beta' + \gamma_2$	48 hr	$\gamma_2 + \beta'$					5 min	$\beta' + \gamma_2$
								15 min	$\beta' + \gamma_2$
								1 hr	$\beta' + \gamma_2 + (\alpha)$
								6 hr	$\alpha + \gamma_2$

475°C		450°C		425°C		375°C		225°C	
Time	Structure	Time	Structure	Time	Structure	Time	Structure	Time	Structure
26.5 hr	$\alpha + \gamma_2$	5 sec	$\gamma' + (\beta')$	5 sec	$\gamma' + \gamma_2$	4 min	$\gamma' + (\beta')$	1 hr	$\gamma' + (\beta')$
		15 sec	$\gamma_2 + \beta' + (\gamma')$	5 min	$\beta' + \gamma_2$	1 hr	$\beta' + \gamma_2 + (\gamma')$	24 hr	$\beta' + \gamma'$
		60 sec	$\gamma_2 + \beta' + (\gamma')$	1 hr	$\beta' + \gamma_2$	26 hr	$\gamma_2 + \beta'$	48 hr	$\beta' + \gamma'$
		2 min	$\gamma_2 + \beta' + (\gamma')$	12 hr	$\beta' + \gamma_2$	187 hr	$\gamma_2 + \beta'$	1 wk	$\beta' + \gamma_2 + (\gamma')$
		5 min	$\beta' + \gamma_2$	60 hr	$\alpha + \gamma_2$				
		10 min	$\beta' + \gamma_2$	148 hr	$\alpha + \gamma_2$				
		16 min	$\beta' + \gamma_2$						
		40 min	$\beta' + \gamma_2$						
		1 hr	$\beta' + \gamma_2$						
		4.5 hr	$\beta' + \gamma_2 + (\alpha)$						
		17 hr	$\alpha + \gamma_2$						
		36 hr	$\alpha + \gamma_2$						

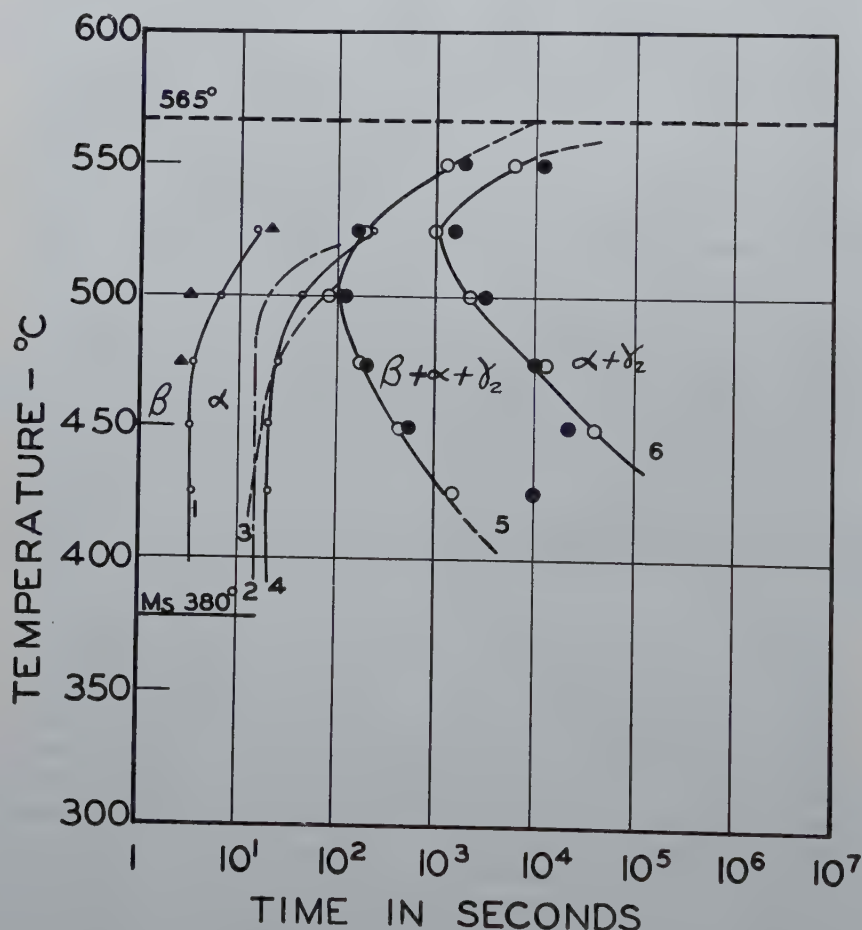


FIG 29—The isothermal transformation diagram for an 11.9 pct Al-Cu alloy.

Curve 1. α precipitation at grain boundaries. $\frac{1}{32}$ in. stock.
 Curve 2. α precipitation at grain boundaries. $\frac{1}{8}$ in. stock.
 Curve 3. α precipitation in grains. $\frac{1}{32}$ in. stock.
 Curve 4. α precipitation in grains. $\frac{1}{8}$ in. stock.
 Curve 5. Beginning of pearlite reaction.
 Curve 6. End of pearlite reaction.
 ▲ Beginning of β' precipitation.

could be expected to decompose precipitating α . This apparently does not take place, rather the γ_2 seemingly

forms at selected sites in the lattice and grows at the expense of the aluminum content of the surrounding β_1 . On the

Table 3 . . . Structures by X ray Analysis in Transformed Specimens of Alloy 11.9 Pct Al

500°C		450°C	
Time	Structure	Time	Structure
60 sec	β'	5 sec	β'
30 min	α, γ_2, β'	30 sec	β'
1 hr	α, γ_2	4 min	$\beta', \alpha, (\gamma')$
		8 min	β', α, γ'
		12 min	β', α, γ'
		32 min	β', α, γ'

425°C	
Time	Structure
77 hr	α, γ_2

depletion of the β_1 to a sufficient degree α forms and in turn grows. By this mechanism the α and γ_2 lattices seemingly are not in contact, but are separated by untransformed β_1 until the final stages of transformation.

An apparent anomaly is accounted for on the basis of this mechanism. This consists in the apparent interchange of role of γ_2 from precipitate structure to matrix structure through the course of the transformations. Thus in Fig 8 it seems that the precipitate structure is γ_2 , while in Fig 9 the "precipitate" structure is α .

It seems that the β'' reaction is comparable to this reaction in the 13.5 pct Al alloy except that the β'' reaction does not go to completion. Thus the β'' formed above about 500°C is for the most part consumed by the pearlite reaction. At lower temperatures increasingly greater proportions of β'' de-

compose to form α , while, as noted before, the pearlite reaction undergoes some important alterations.

The β'' structure probably does not differ from the matrix structure in so far as ordering is concerned. Rather from the fact that α forms from β'' it appears reasonable to conclude that β'' is aluminum dilute. A quantitative evaluation of the β'' crystal structure is out of question with the X ray diffraction techniques used in this investigation, but it has appeared that β'' has essentially the same structure as β' .*

Summary

1. It has been shown that the pearlite reaction is not found at compositions appreciably away from eutectoid composition on isothermal transformation of β -CuAl alloys. This reaction cannot be considered as generally typical of eutectoid transformations.

2. In agreement with previous work^{2,4} it has been found that the pearlite reaction exists in the alloy of eutectoid composition. It has been found, however, that the interlamellar spacing of this pearlitic structure first decreases and then increases with decreasing transformation temperature. This behavior was wholly unexpected and cannot be accounted for on the basis of current notions of the pearlite reaction.

3. A bainite type reaction or, for short, a bainite reaction has been found in the CuAl alloys studied. The first reaction in the 13.5 pct Al alloy and the β'' reaction in the 11.9 pct Al alloy are placed in this category. In both of the alloys studied the β_1 matrix forms zones enriched and depleted in aluminum. For the 13.5 pct Al alloy the enriched zones decompose to form γ_2 which grows by an accretion process. The depleted zones in turn form α which also grows by an accretion process. The formation of α occurs at a much later time than the formation of γ_2 . The β'' transformation in the 11.9 pct Al alloy differs from this mainly in that α precipitates before γ_2 .

Acknowledgment

Of the materials used in preparing the alloys studied, the OFHC copper was supplied by The American Metal Co. Ltd., New York, while the high purity aluminum was furnished by The Aluminum Company of America.

*It is evident that this discussion pertains to the structures of the quenched alloys.

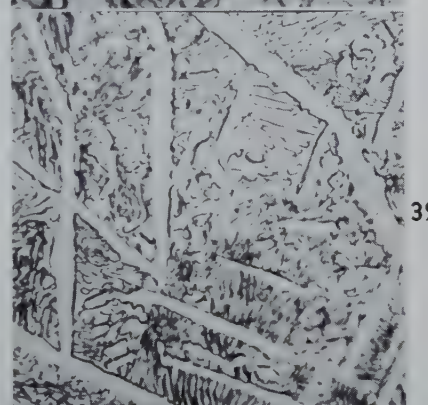
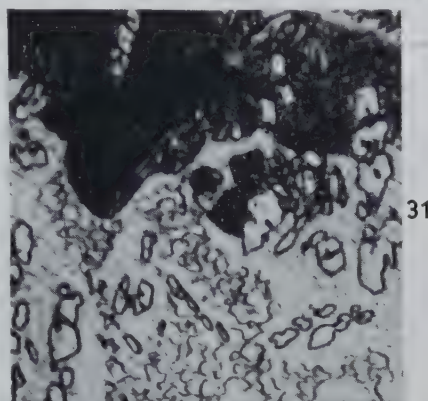
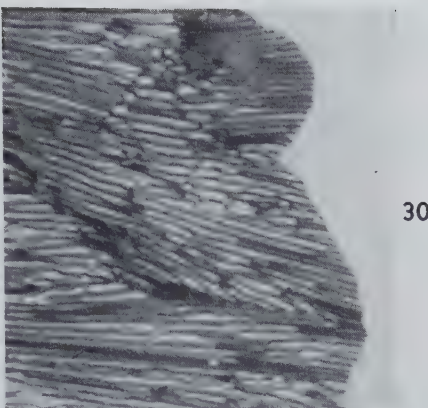


FIG 30—11.9 pct Al-Cu specimen transformed at 550°C for 30 min. $\frac{1}{32}$ in. stock. $\times 750$

FIG 31—11.9 pct Al-Cu specimen transformed at 500°C for 8 min. $\frac{1}{32}$ in. stock. $\times 1000$

FIG 32—11.9 pct Al-Cu specimen transformed at 425°C for 77 hr. $\frac{1}{32}$ in. stock. $\times 1000$

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Compression Textures of Copper and Its Binary Alpha Solid Solution Alloys*

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Previous investigations have shown that the cold rolling textures^{1,2} and the drawn wire textures³ of copper change their secondary components after the addition of about 1 pct aluminum and 5 pct zinc, but do not change after the addition of as much as 30 pct nickel. However, few data are available on compression textures. Barrett⁴ reports from unpublished work with E. L. Layland that compression rolled copper develops a texture which is predominately {110} in the compression plane with a secondary spread to {113} and weaker spread to {100} as shown in Fig 1. However, 70-30 alpha brass similarly treated develops a texture again predominately {110} in the compression plane and with a weak secondary spread to {113} but with moderate {111} secondary intensities illustrated in Fig 2.

Experimental Procedure

Specimens of the compositions shown in Table 1 remaining from a previous investigation³ were annealed to a grain size of 0.090 mm average diam and machined to cylinders 0.20 in. diam and 0.20 in. high. Cylinders were compressed 90 pct reduction in height between lubricated steel blocks in a tensile machine. Glancing X ray photographs using copper K-alpha radiation were taken at an angle of θ degrees to the compression surface. {111}, {002} and {220} rings were analyzed to define the textures shown in Table 1.

The results are in agreement with Barrett⁴ as to types of textures and are essentially in agreement with previous work^{1,2,3} as to the effects of composition. In the wire study³ it was reported that the 4.99 pct zinc alloy had a brass-type texture, while the data in Table 1 indicate a copper-type texture for compression. The wire specimen of the 4.99 pct zinc alloy was re-examined by X rays and it was established that the {100} component still exists in the wire axis. Therefore, Table 2 of Ref. 3 is in

error and should indicate a double [111], [100] copper-type texture for this alloy. This correction results in complete consistency with regard to compositional effects, namely that copper and its alloys containing up to approximately 1 pct aluminum, 5 pct zinc and at least 30 pct nickel§ develop a copper-type texture and more concentrated copper-aluminum and copper-zinc solid solution alloys will form a brass-type texture.

Summary

Compression textures developed in copper and its alpha solid solution alloys of aluminum, nickel and zinc are essentially the same types as those re-

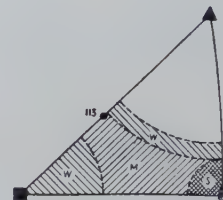


FIG 1—Orientations in copper compressed 97 pct by compression rolling (Barrett).

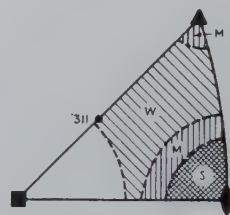


FIG 2—Orientations in 70-30 brass compressed 97 pct by compression rolling (Barrett).

Table 1 . . . Composition and Texture

Material	Composition		Texture of Compression Plane				
	Wt. Pct	At. Pct	{110}	{111}	{100}	{113}	Fig
Copper*			S				1
Cu-Ni.....	30.70	32.44	S		W	M	1
Cu-Al.....	0.94	2.16	S	W	W	M	Transition between 1 and 2
	3.69	8.28	S	M		W	2
	7.76	16.58	S	M		W	2
Cu-Zn.....	4.99	4.83	S		W	M	1
	10.16	9.90	S	M		W	2
	12.42	12.12	S	M		W	2
	29.22	30.00	S	M		W	2

* Phosphorous deoxidized electrolytic copper
Note: S = strong, M = medium and W = weak

ported by Barrett⁴ with compositional effects occurring at approximately the same percent additions as previously reported for rolled sheet^{1,2} and drawn wire.³

§ Higher nickel alloys were not investigated.

Acknowledgment

The Scovill Manufacturing Co., Waterbury, Conn., furnished the copper and brass alloys, performed certain chemical analyses and supplied laboratory facilities where needed.

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Technical Note No. 18E. Manuscript received April 25, 1949.

* Some of the data included in this paper are from a report presented to the Faculty of the School of Engineering, Yale University, by Delmar E. Trout, II, in candidacy for the Degree of Master of Engineering, May 1948.

† Assistant Professor of Metallurgy, Yale University, New Haven, Conn.

‡ Scovill Manufacturing Co., Waterbury, Conn.

§ References are at the end of the note.

Surface Orientation and Rolling of Magnesium Sheet

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Magnesium alloy sheet has less ability to accept bending at room temperature than most of the heavier metals. In work designed to improve the bend properties, the preferred orientation of the sheet is of major importance as it is in all studies of the properties of wrought magnesium products. When rolled into sheet, all of the common magnesium alloys form an orientation texture having the basal (002) planes approaching parallel to the surface of the sheet. This texture is only slightly affected by annealing. Magnesium single crystals are highly anisotropic, and, as might be expected, so are magnesium alloy wrought products in which a strong preferred orientation is developed. It is therefore not surprising that bend properties are affected by orientation. Ansel and Betterton¹ reported that the orientation of AZ31X† sheet varies from surface to center and that bend properties are improved by etching away the sharply oriented material at the surface of the sheet to reach the more broadly oriented structure below. This paper covers a study of that orientation, either during the rolling process or by treatment of the finished sheet, in an effort to improve the bend properties and toughness of sheet.

Literature

The orientation texture of magnesium and magnesium alloy sheet has been studied extensively. Early determinations² showed that pure magnesium sheet has a preferred orientation in which the basal planes are parallel to the sheet surface within very narrow limits. J. C. McDonald³ and J. D. Hanawalt⁴ reported that sheet containing a small amount of calcium develops a "double" texture, that is, the majority of the basal planes are a few degrees from parallel to the surface and there is a noticeable vacancy at the parallel position. Bakarian⁵ made careful quantitative pole figures of both pure magnesium sheet and MI alloy

sheet which show these features. In all of these studies, however, the orientation was determined by transmission methods in which the resulting pattern is an average through the thickness of the sheet.

The tendency of wrought metal to exhibit a different orientation at the surface from that in the center has been reported by many investigators. G. von Vargha and G. Wasserman⁶ found that with copper, aluminum, iron, and brass the textures of rolled compared to drawn wires were the same at the center but differed markedly at the surface. It was also reported by investigators⁷ that the orientation of rolled aluminum varies from surface to center. Hargreaves⁸ found that the surface texture of AM503 (magnesium alloy similar to MI) sheet was different from the center texture. It is reported by Edmunds and Fuller⁹ that zinc alloy sheet sometimes had a thin layer at the surface with a strong orientation of the basal planes parallel to the surface, which, if present, impaired the bend properties of the sheet.

Part I Surface Orientation of Magnesium Alloy Sheet and the Effect on Properties

Attempts to correlate the bend properties of magnesium alloy sheet with tension ductility over short gauge lengths proved unsuccessful and the subsequent investigation showed that nonuniformity in orientation is a con-

tributing factor as the properties of the surface material have a much more important effect in bending than in tension. A program to study the relationship between surface orientation at the surface and bend properties was then undertaken.

First, the effect of etching away the surface of sheet on the bend properties and the orientations at the various depths were studied. Sheet samples of MI, AZ31X, and AZ61X were etched in dilute nitric acid to remove the surface material for various depths to 0.015 in. As may be seen in Table 1, the minimum bend radius improved considerably as the surface layers were etched away but it was necessary to etch the sheet quite deeply, much more so than was found necessary by Edmunds and Fuller⁹ on zinc sheet. It is also apparent that the amount of etching required is a function of the sheet thickness. In all of this work, radii were measured as R/t , the radius divided by the sheet thickness, in order to eliminate the effect of the reduction in sheet thickness produced by the etching.

To determine the orientation texture of the sheet, X ray reflection patterns were taken using copper radiation with the beam striking the specimen at an angle of 17° to the surface, which is the Bragg angle for the (002) planes of magnesium. Two exposures were made of each specimen, one with the beam perpendicular to the rolling direction and the other with the beam parallel to the rolling direction. The symmetry of the preferred orientation in magnesium sheet is such that these two photographs gave an approximation of the pole figure sufficiently accurate for qualitative work and it was not thought worthwhile to make complete pole figures.

These X ray patterns show that the orientation has a much narrower spread at the original surface of the sheet than below the surface. The narrow spread is found in sheet having the majority of the basal planes (002) parallel to the surface, and since this is an unfavorable position for slip, it is

Cleveland Meeting, October 1949.
TP 2657 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received April 27, 1949.

* The Dow Chemical Co., Midland, Michigan.

† The A.S.T.M. system of alloy designation is employed throughout this paper. AZ31X is Mg + 3%Al + 1%Zn; AZ61X is Mg + 6%Al + 1%Zn; AZ80X is Mg + 8%Al + 0.5%Zn; MI is Mg + 1.5%Mn + 0.10%Ca. X indicates high purity; h, hard rolled sheet, and a, annealed sheet. In addition, AZ31X when in sheet form contains 0.13%Ca.

† References are at the end of the paper.

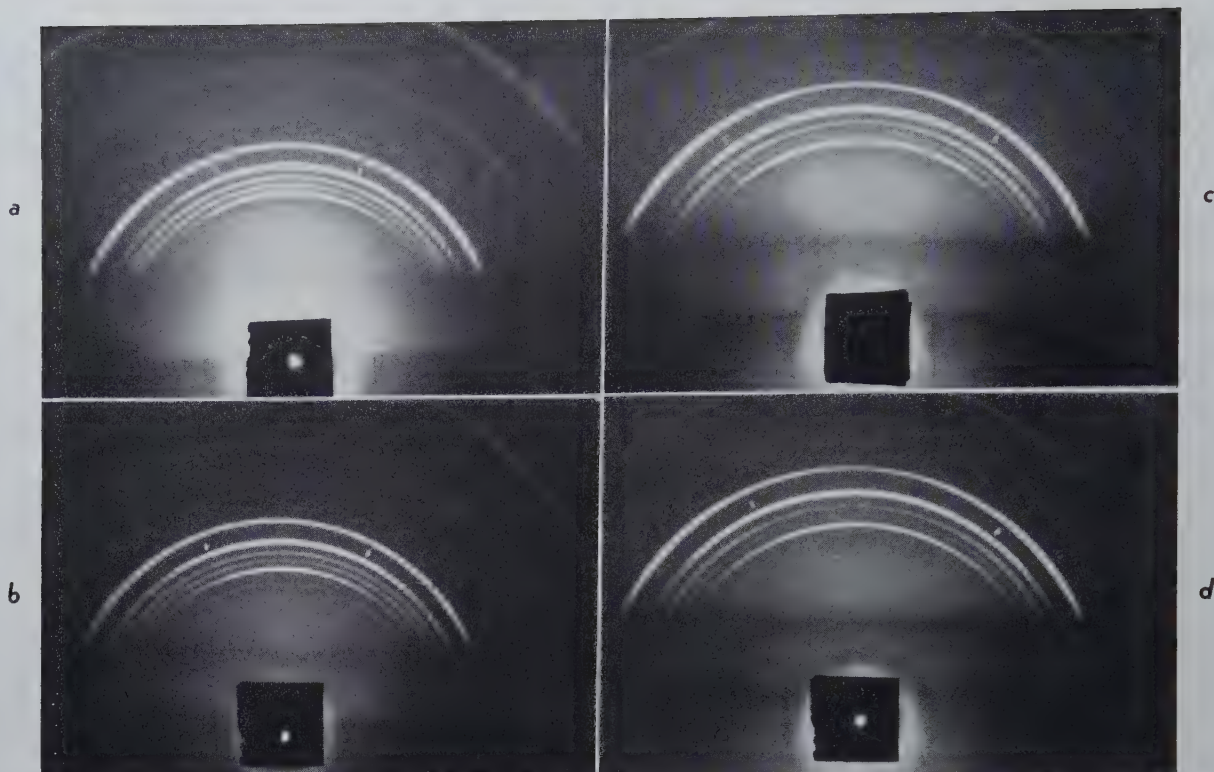


FIG 1—Effect of Etching on Surface Orientation of 0.091 in. AZ31X-h Sheet.

Original Surface (a) X ray beam perpendicular to rolling direction, (b) beam parallel; etched 0.015 in. (c) beam perpendicular and (d) beam parallel. Copper radiation. Dots are placed to show limits of maximum intensity of (002) reflections.

Table 1 . . . Effect of Etching on Bend Radius of Mg Alloy Sheet

Alloy	Original Gauge Inches	Minimum Successful Bend Radius (r/t) Etch in. in. per Side				
		None	0.001	0.002	0.005	0.010
AZ31X-h	0.040	9.7	7.8	5.7	5.7	
AZ31X-h	0.064	6.4	6.0	5.8	5.8	
AZ31X-h	0.091	6.4	6.3		5.7	4.8
AZ31X-a	0.091	3.3	2.6			2.2
M1-a	0.040	4.4	4.5	3.9	2.4	3.5

not surprising that removing this layer improves the bend properties.

In the reflection patterns of 0.091 in. AZ31Xh sheet at the original surface and after etching 0.015 in. (Fig 1), the difference in spread is quite noticeable. The minimum bend radius was $6.4t$ before etching and $4.8t$ after etching. Other samples gave the same correlation, the bend properties improve as the orientation spread at the surface increases.

Tests made to determine the depth of penetration of the X ray beam in this type of reflection photograph showed that by use of copper radiation the beam penetrates to a depth of approximately 0.003 in. before its intensity is reduced to an ineffective level. The patterns must be interpreted as an integrated pattern of a layer 0.003 in. thick but heavily weighted in favor of the extreme surface. Subsequent tests showed that iron radiation, used for much of the later work, limited the depth of penetration to about 0.002 in.

EFFECT OF ETCHING ON TENSILE PROPERTIES

Although etching away the surface layers of sheet produces a marked improvement in bend properties, it was found that there is only an insignificant change in tensile properties. Tests on etched specimens showed a slight tendency for the etching to improve the elongation and lower the yield and tensile strengths; but the decrease is so slight as to be within normal tensile test variations and is only visible as a trend. This result is very reasonable, for, in a tension test, the entire cross-section of the sample is tested, whereas in bending the maximum elongation is found only in the extreme surface fibers and hence the minimum bend radius is determined by the elongation possible at the surface.

ETCHING EXTRUDED STRIP

The effect of etching on the bend properties of sheet aroused interest in

the behavior of extruded strip having a similar orientation to sheet when a transmission pattern is taken. Extruded strips, $\frac{1}{8}$ in. by $\frac{3}{8}$ in., of AZ80X, AZ61X, M1, and AZ31X were etched in the same manner as the sheet with rather startling results. Etching AZ31X strip improved the bend properties as in the sheet, but AZ80X and AZ61X strip had poorer bend properties after etching and M1 strip showed no effect. Diffraction patterns by reflection from the surface showed that the surface orientations of the various samples correlated with their bend properties. In the predominate orientation at the surface, the basal planes are not parallel to the surface, but are inclined to it at an angle of 10° to 30° .

Table 2 shows the effect of etching 0.015 in. on the bend properties of AZ80X and M1 strip and the corresponding surface diffraction patterns are shown in Fig 2. As can be seen readily, the orientation of AZ80X at a depth of 0.015 in. has a much narrower spread than at the surface, whereas in M1 strip there is little difference. Crystallites having the basal plane at an angle to the surface are in a more favorable position for basal slip than if parallel; therefore, the greater the percentage of the material at the surface in a favorable position, the better the bend properties. The results of the experiments on extruded strip confirm

a

c

b

d

FIG 2—Effect of Etching on Surface Orientation of Extruded $\frac{1}{8}$ in. X 3 in. Strip.

(a) AZ80X as extruded and (b) Etched 0.015 in.

(c) M1 as extruded and (d) Etched 0.015 in.

Copper radiation, beam perpendicular to extrusion direction.

the importance of surface orientation in determining bend properties.

SHOT PEENING

Having determined the importance of a favorable surface orientation in obtaining good bend properties in sheet, the problem of improving that orientation presents itself. There are two general ways in which this can be done; one is to alter the orientation of the rolled sheet, the other is to control the orientation during the rolling process.

One of the methods of altering the surface of the finished sheet is to etch away the unfavorably oriented material at the surface but because of the large amount of metal wasted by this process it is not commercially feasible. Shot peening offers a means of working the surface which utilizes flow characteristics entirely different from rolling. It was found that when AZ31Xa sheet was shot peened severely and then annealed at 600°F (310°C), a random orientation at the surface could be produced (Fig 3). Metallographic examination showed the affected layer to be about 0.003 in. deep. By this treatment the minimum bend radius was reduced from 3.5*t* to 2.5*t*.

REVERSE BENDING

A more effective and practical way to alter the surface orientation was

Table 2 . . . Effect of Etching on $\frac{1}{8}$ in. Extruded Strip

Alloy	Etch	Min. Bend Radius	* Surface Orientation, Arc of Max. Intensity
AZ80X	None	3.0 <i>t</i>	+10°, +54°
AZ80X	0.015 in. per side	4.0 <i>t</i>	-10°, +10°
M1	None	4.0 <i>t</i>	+3°, +29°
	0.015 in. per side	3.9 <i>t</i>	+5°, +22°

* Orientation is spread of basal planes in extrusion direction from parallel to surface.

found when it was learned that bending the sheet to about 15*t* radius through rolls, straightening, bending in the opposite direction and annealing produce a radical change in surface orientation and improve the bend properties. If the process is repeated a second time there is further broadening of orientation and further improvement in bend properties, but additional repetition is not effective. As is shown by the data in Table 3, AZ31X sheet had a 3.5*t* bend radius in the annealed condition, but if treated by reverse bending the minimum radius was reduced to 2.5*t* after the first treatment and 1.2*t* after the second. There is also an accompanying loss in yield strength, an increase in elongation, and a substantial increase in toughness.

The reverse bending process was studied in detail by X ray methods and it was found that the major orientation

change occurs during annealing, probably as a result of the twinning which takes place on the compression side of the bend. This is one of the few known cases in which annealing magnesium results in an appreciable change in orientation.

Roller leveling is somewhat similar to this reverse bending and has been cited in the literature¹¹ as a way to improve bend properties, but attempts to use this method were unsuccessful even when the two stage anneal was used. The difference is in the severity of the bends since the radii must be small enough to produce strong twinning on the compression side of the bend.

Part 2 The Control of Surface Orientation in the Rolling Process

Although the surface orientation of magnesium alloy sheet can be altered by etching, shot peening, or reverse bending, it is, of course, much more economical to roll in such a manner as to secure the desired properties without further treatment. A comprehensive program was undertaken to study the effect of hot and cold rolling variables on the surface orientation and properties of AZ31X alloy, the most widely used magnesium sheet alloy. The

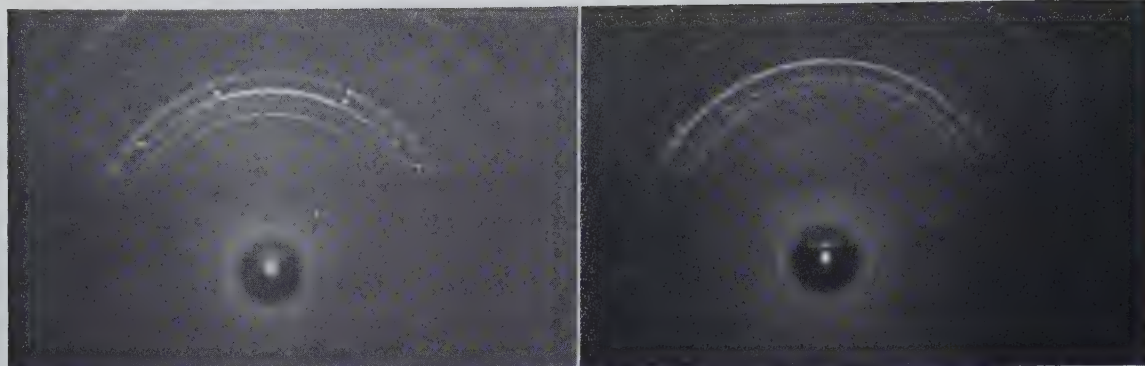


FIG 3—Effect of Shot Peening on Surface Orientation of 0.064 in. AZ31X-a Sheet.
(a) Original Surface (b) Shot peened and annealed. Copper radiation.

Table 3 . . . Effect of Reverse Bending in Both Directions on AZ31X-h

Treatment	Tensile Properties			Minimum Bend Radius
	Ultimate psi	0.2 Pct Yield psi	Pct E in 2 in.	
Original + 600°F Anneal.....	37,800	23,000	21	3.5 <i>t</i>
Reverse Bend + Anneal.....	37,300	20,800	23	2.5 <i>t</i>
(Reverse Bend + Anneal) × 2.....	35,600	15,900	24	1.2 <i>t</i>

variables of temperature, reduction per pass, lubrication, and total reduction were studied as well as the effect of different combinations of them. The minimum bend radius, tension and compression strengths, and toughness gave good correlation with the resulting orientation.

EQUIPMENT

All laboratory investigations were carried out on a laboratory 2-Hi rolling mill having 8 in. diam by 12 in. rolls, and, in addition, a few pilot plant experiments were carried out on a 2-Hi mill having 32 in. by 60 in. rolls. Orientation was determined by X ray surface reflection using both copper radiation and iron radiation.

HOT ROLLING

Most of the experimental work was done on hot rolling as it was found that more could be accomplished in controlling the orientation in hot rolling even when the sheet was finished by cold rolling. The hot rolling variables of reduction per pass and temperature were studied by rolling AZ31X from 0.250 in. to 0.082 in. in four ways:

1. 800°F, (430°C), 20% reduction per pass
2. 800°F, 5% reduction per pass
3. 500°F, (260°C), 20% reduction per pass
4. 500°F, 5% reduction per pass

The resulting hot rolled material was then annealed at 700°F (370°C), cold rolled 20 pct, and final annealed at 275°F (135°C). Surface orientation and

properties were determined at each stage of the process. The higher temperatures and heavier passes produced a sheet having a much broader spread in the orientation of the basal plane at the surface. This sheet had better bend properties and, in addition, a considerably higher compression yield strength. The effect of the hot rolling conditions carried through the subsequent finishing operations of annealing and cold rolling. The properties of the sheet before and after cold rolling are given in Table 4. Surface reflection X ray pictures of the finished sheet are shown in Fig 4 with the differences in spread of the basal reflection evident.

The most probable explanation for the temperature effect is that the relative activity of the slip systems is altered by increasing temperature. There has been some indication that this may be true^{2,12,13} but no adequate proof exists. It is interesting to note Kolesnikov's¹⁴ report that three different textures were produced when zinc sheet was rolled at 20°C (70°F), 150°C (300°F), and 380°C (720°F). There are two possible reasons for the effect of reduction per pass; it may be the result of more uniform working through the thickness of the sheet or it may be the result of stress changes as discussed under lubrication. McLachlan and Davey¹⁵ found that with iron-nickel alloys, the orientation of a 0.001 in. surface layer is affected by reduction per pass but below this there is no change. Anderson and Kehl,¹⁶ on the other hand, found that with copper the reduction per pass does not influence either the surface or the overall texture.

ROLL LUBRICATION

Lubrication during hot rolling should have an effect on the stress system and possibly on the surface orientation. To study this variable, sheets were hot rolled from 0.250 in. to 0.075 in., annealed at 700°F (370°C), and cold rolled to 0.064 in. Rolling was done with palm oil on the rolls and with dry rolls. In the tests reported in Table 5, when lubricant was used for hot rolling it was also used for cold rolling; if hot rolling was dry, cold rolling was also dry. The use of lubricant causes a marked improvement in the bend properties of AZ31X but in the hard rolled condition the greatest improvement is secured if the sheet is subsequently etched to remove 0.0005 in. per side. The only explanation that can be offered for the results of etching is that even with lubrication there is a very thin layer of material with poor orientation at the surface of the sheet. The X ray method is, however, not sufficiently sensitive to prove or disprove this theory. The wider surface orientation resulting from the use of a lubricant is believed to be the result of reduction of the tensile stress on the surface due to the friction of the rolls. Experiments by the author on M1 alloy single crystals have shown that slip and fracture characteristics are different in the rolling process from those in pure compression. Single crystals having the basal plane within 5° of parallel to the rolling surface could be hot rolled successfully but fractured when subjected to compression in a testing machine operated at the same temperatures.

The use of adequate lubrication is believed to cause the stress system at the surface to approach more nearly the stresses found in the center of the sheet and, therefore, to reduce the variation in orientation from surface to center.

X ray pictures illustrating the effect of lubrication on the surface of AZ31X

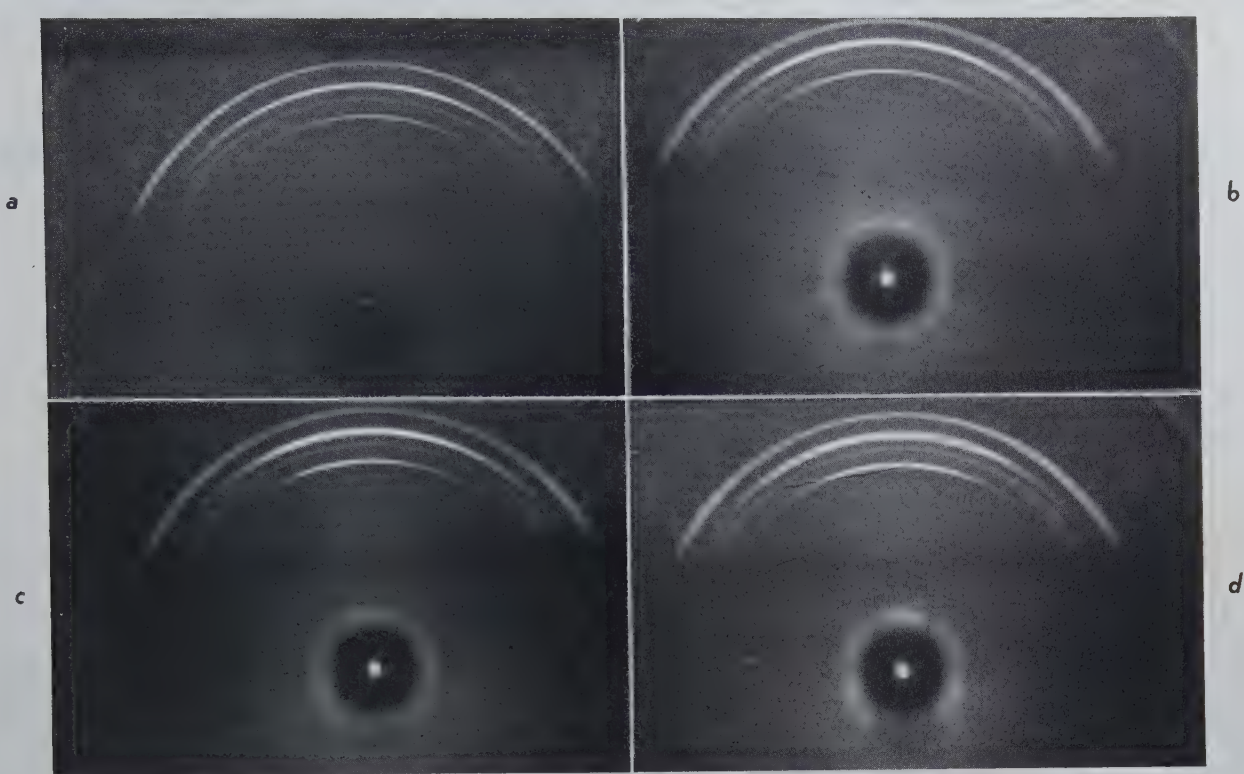


FIG 4—Effect of Hot Rolling Temperature and Reduction per Pass on Surface Orientation of Finished 0.064 in. AZ31X-h Sheet.

All sheet was annealed, cold rolled 15 pct and final annealed at 275°F (135°C)
Copper radiation, beam perpendicular to rolling direction.

(a) Hot rolled 800°F, 20 pct per pass (b) 800°F, 5 pct per pass (c) 500°F, 20 pct per pass (d) 500°F, 5 pct per pass

sheet finished by annealing and cold rolling 15 pct with dry rolls are shown in Fig 5. Further investigation shows that for best results lubrication should be confined to hot rolling, since when used for cold rolling it does not improve the resulting orientation and lubrication during cold rolling is well known to reduce the capacity of magnesium alloys to withstand cold rolling without cracking.^{1,17}

REQUIRED TOTAL REDUCTION IN HOT ROLLING

Having established that surface orientation could be altered by hot rolling conditions, tests were run to determine the minimum amount of hot rolling required under optimum conditions to secure the desired surface orientation and bend properties. These tests showed that the conditions under which the last 30 to 40 pct of the reduction in hot rolling is carried out determines the final properties.

PERSISTENCE DURING COLD ROLLING

All the data presented so far were obtained on sheet finished with 15 to 20 pct cold roll. It is surprising that the orientation produced in hot rolling persists so strongly through annealing and cold rolling operations. It would seem

Table 4 . . . Effect of Hot Rolling Temperature and Reduction per Pass AZ31X Alloy

Hot Rolling		Cold Roll†	Anneal °F	Min. Bend (r/t)	Tensile*	Yield*	Pct E in 2 In.	Comp. Yield*
Temp. °F	Reduction per Pass Pct							
800	20	None	700	3.5	37,100	21,400	21	15,700
800	5	None	700	5.0	35,400	21,400	17	11,900
500	20	None	700	6.5	38,200	25,200	21	14,900
500	5	None	700	6.0	37,200	23,600	18	11,600
800	20	20 %	275	9	44,400	35,700	9	33,000
800	5	20 %	275	9	41,600	32,800	7	28,700
500	20	20 %	275	14	45,300	35,700	7	32,200
500	5	20 %	275	14	42,400	34,100	3	28,200

* Psi.

† Annealed 1 hr 700°F before cold rolling.

probable that sufficient reduction in cold rolling would remove any influence of hot rolling conditions. Thin gauge magnesium alloy sheet is produced commercially by multiple cold rolling from a minimum hot rolled gauge of 0.075 in. Total reductions of 20 to 40 pct with intermediate anneals are used. The tests conducted in the laboratory consisted of cold rolling two batches of sheet, one of which had been hot rolled with lubricated rolls at 800°F, 20 pct per pass, and the other had been hot rolled with dry rolls at 600°F, 20 pct per pass. Two successive cold rolls of 20 pct each followed by one of 30 pct were used, annealing at 700°F (370°C) between cold rolls. The superior bend properties of the first batch persisted in the hard rolled sheet

through the entire cold rolling. It did, however, become slight after the second cold roll. In sheet final annealed at 600°F (320°C) all effect of the hot rolling conditions on bend properties was lost after the second cold roll. The sheet hot rolled at the higher temperature maintained a slightly better compression yield strength through the entire series of cold rolls.

COLD ROLLING

In addition to the work on hot rolling, the effects on surface orientation of roll lubrication, roll temperature, reversing the direction of each pass, reduction per pass, and total reduction in cold rolling were studied. The differences obtained were much less important than in the studies on hot rolling. It

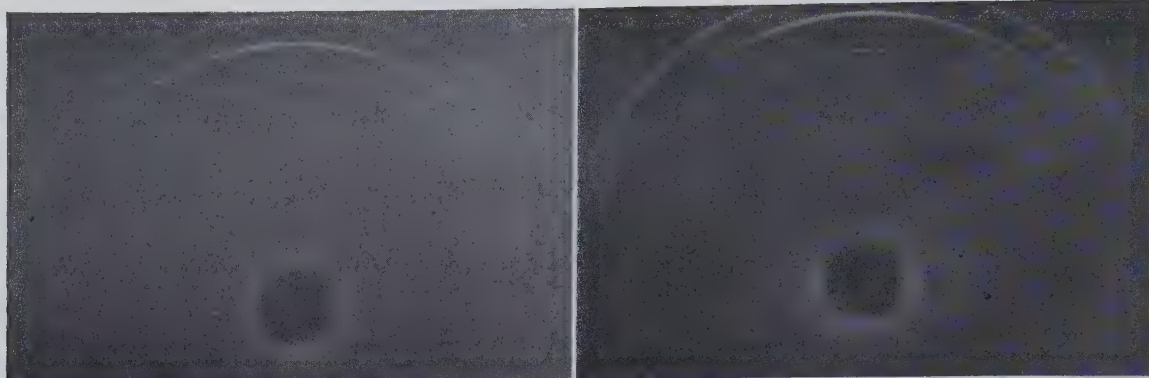


FIG 5—Effect of Lubrication During Hot Rolling on Surface Orientation of Finished 0.064 in. AZ31X Sheet.

(a) Palm oil on rolls, (b) rolls dry.
All sheet hot rolled at 800°F (430°C) and 20 pct per pass.
Sheet was annealed at 700°F (370°C) and cold rolled 15 pct with dry rolls.
Iron radiation perpendicular to rolling direction.

Table 5 . . . Effect of Lubrication during Rolling AZ31X Sheet

Hot Rolling*			Anneal °F	Min. Bend Radius (r/t)		Ulti- mate†	Yield†	Pct E in 2 In.	Comp. Yield†
Temp. °F	Red. per Pass Pct	Lubri- cant		No Etch	Etch 0.0005 in. per Side				
800	20	Oil	275	6	3.5	41,500	33,900	10	31,000
800	20	Dry	275	6.5	6	42,000	34,700	9	29,600
550	5	Dry	275	14	12	38,100	32,300	3	25,500
800	20	Oil	600	1.5	1.5	36,400	26,200	21	18,900
800	20	Dry	600	2.5	2.5	37,200	23,300	20	18,900
550	5	Dry	600	3	3	37,400	23,000	18	16,400

* All sheet annealed 700°F (370°C) and finished by cold rolling 15 pct to 0.064 in. gauge before final anneal.
† Psi.

was found that the best combination of strength and ductility in hard rolled sheet is produced by the present commercial practice of finish cold rolling 20 to 30 pct followed by a low temperature anneal at 325°F (160°C) rather than finishing with a small cold reduction without final anneal.

WARM ROLLING

As an alternative to finish cold rolling in securing the desired surface and properties, warm rolling is sometimes used. Tests on sheet finished by warm rolling at 300°F (150°C) and 400°F (200°C) showed poorer bend properties than on sheet finished by cold rolling.

OTHER MAGNESIUM ALLOYS

The work on rolling was not extended to cover magnesium alloys other than AZ31X since this alloy accounts for by far the largest quantity of production sheet, and is used almost entirely for those applications in which room temperature formability and toughness are critical factors. Some laboratory work has shown that the hot rolling temperature affects the cold rollability of AZ31X both with and without calcium and also that of AZ61X.

Summary

1. Studies of the crystallographic orientation of magnesium alloy sheet show that the spread in the orientation

of the basal plane is considerably less at the surface than in the center of the sheet.

2. A narrow orientation at the surface is detrimental to the bend properties of the sheet.

3. The layer having a narrow orientation can be removed by etching, or the orientation can be broadened by shot peening or reverse bending with a resultant improvement in bend properties.

4. The surface orientation of AZ31X sheet can be improved by higher temperature, higher reduction per pass, and adequate lubrication during hot rolling with a resultant improvement in bend properties and a slight improvement in compression yield strength.

5. The improved surface orientation resulting from hot rolling persists to a remarkable degree through subsequent annealing and cold rolling operations.

6. Variations in cold rolling have only a very slight effect on surface orientation.

Acknowledgment

The author wishes to acknowledge the assistance of J. B. Hess who did all the very considerable X ray work involved in this program.

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Recrystallization Texture and Coarsening Texture in High Purity Aluminum

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Introduction

It has been known for many years that in cold drawn polycrystalline aluminum the recrystallization texture is practically identical with the deformation texture.^{1,2,3} V. Goeler and Sachs⁴ stated that in cold rolled polycrystalline aluminum, too, the deformation texture is retained upon recrystallization. This behavior was interpreted by Dehlinger⁵ as an indication that each recrystallized grain inherits the orientation of the matrix in which it grew. However, Burgers and Basart showed⁶ that in deformed aluminum single crystals the orientation of the recrystallized grains strongly deviates from that of the matrix. Barrett demonstrated⁸ that even in deformed polycrystalline aggregates the local orientation within each deformed grain changes on recrystallization. No explanation has as yet been offered of how the aggregate texture can be retained, while the local texture is everywhere changing.

According to Burgers and Louwse⁶ the orientation change on recrystallization in compressed and rolled aluminum single crystals corresponds to a rotation around [112] axes lying in the active slip planes and perpendicular to active slip directions. This orientation change was rationalized by them on the assumption that the orientation of the recrystallized grains is determined by the orientation of the available nuclei, and that these nuclei are small fragments of the deformed crystal, rotated in the deformation process. Barrett pointed out,⁷ however, that these assumptions were by no means proven, and that the total volume of material necessary for the nuclei is so small, that it must be available in any orientation whatever.

It is, therefore, unlikely that an "oriented nucleation" theory could satisfactorily explain the orientation changes in recrystallization of a deformed crystal. Furthermore, in contradiction to Burgers and Louwse's conclusions, Barrett's results for the orientation change could be best expressed in terms of rotations around [111] axes. Thus agreement is lacking not only in the interpretations but apparently even in the experimental results contributed by the various investigators.

The orientation relationships on coarsening* in a highly oriented polycrystalline matrix were first investigated by Burgers and Basart.⁶ They produced a fine grained highly oriented matrix by the recrystallization of a compressed or rolled single crystal of

* Just as the recrystallized grains grow at the expense of the deformed matrix, and finally replace it, under suitable conditions the recrystallized grains may be in turn replaced by a new generation of grains, usually many times larger than the recrystallized grains and growing at their expense. The growth of these very large grains may be designated as discontinuous grain growth or coarsening. (The following other terms have been used by various authors: abnormal grain growth, exaggerated grain growth, germination, and secondary recrystallization. These terms are deemed to be unsuitable, as they have been used indiscriminately to describe the formation of large grains by various mechanisms.¹¹)

Recent work indicates^{10,11} that an essential condition for coarsening is that in a fine grained structure continuous or gradual growth be impeded. There are at present two mechanisms known, each one of which is capable of accomplishing this, namely the inhibition by a dispersed phase and the restraint of growth resulting from a strongly developed texture. Correspondingly, one may speak of "inhibition-dependent coarsening" or "texture-dependent coarsening." In metallurgical practice intermediate cases often occur, where gradual grain growth is impeded by a combination of the two mechanisms mentioned.

Cleveland Meeting, October 1949.
TP 2675 E. Discussion of this paper
(2 copies) may be sent to *Transactions*
AIME before December 1, 1949.
Manuscript received June 6, 1948.

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¹ References are at the end of the paper.

aluminum, and studied the orientation relationships in three successive generations of crystals: the "deformation texture" in the single crystal, the recrystallization texture, and the coarsening texture. Burgers²⁴ showed that the preferred orientation of each successive generation was almost always widely different from that of the preceding one, but that the coarse grains often approximately reverted to the orientation of their grandparent, the deformed single crystal. This observation, and the idea that the orientation of the coarse grains is determined by that of the available "nuclei," led to the concept that the coarse grains are nucleated by the remnants of the deformation texture.^{12,15} On the other hand, Bowles and Boas¹⁵ found, in a carefully conducted experiment, that in recrystallized fine grained copper of cube texture coarse grains grow in orientations which can be derived from the orientation of the recrystallized matrix by rotation around a [111] direction; the coarsening texture here does not appear to be related in any simple manner to the original rolling texture from which the cube texture had been obtained by recrystallization.

It may be assumed that in a recrystallized material, even with a strong texture, there always are present some grains of practically any orientation. These may serve as "nuclei" for the coarse grains, provided that the conditions are favorable for their growth. The existence of a coarsening texture may be considered as an indication that grains with certain relative orientations with respect to the preferred orientation of their neighbors can grow much faster than others. This "oriented growth" theory was briefly suggested by van Arkel, as early as 1936,¹³ and it was mentioned by Burgers.¹⁴ Recently, C. G. Dunn produced direct

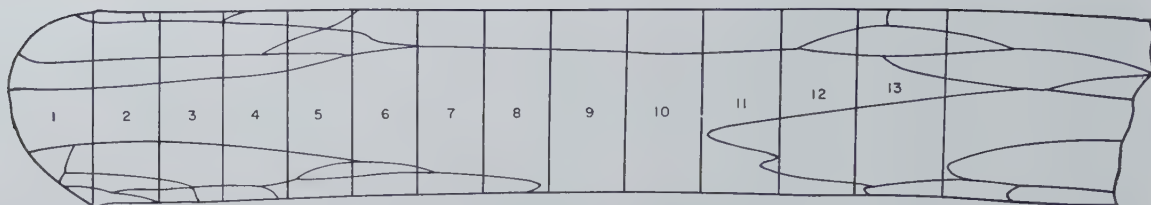


FIG 1—Outline of grain boundaries in rolled high purity aluminum bar, containing crystal I (large grain in center).

Location of saw-cuts is indicated by straight cross lines. Specimens are numbered 1 to 13.

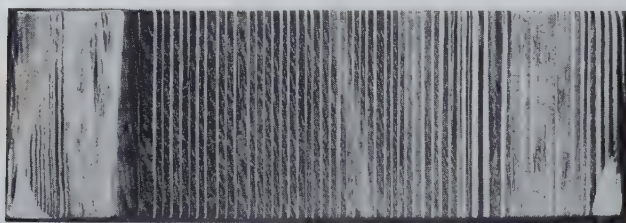


FIG 2—Spec. 7 of crystal I, as rolled, showing deformation bands in direction of rolling.

Deep etched. Magnification: $\times 3$. Reduced one half in reproduction.

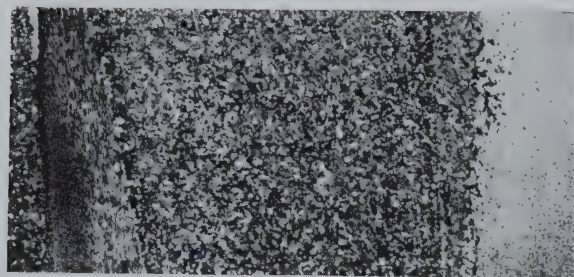


FIG 3—Spec. 4 of crystal I, rolled and annealed 25 min. at 400°C, showing fine grained recrystallized structure.

Deep etched. Magnification: $\times 4$. Reduced one half in reproduction.

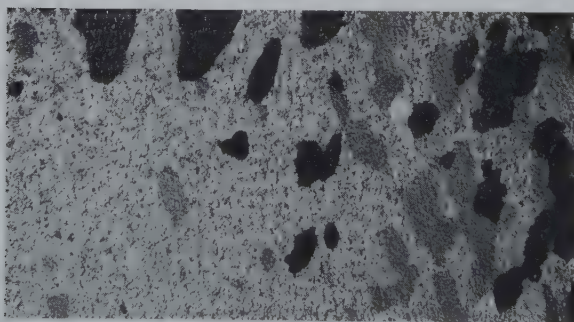


FIG 4—Spec. 8 of crystal I, rolled and annealed 745 min. at 400°C, showing several coarse grains in fine grained matrix.

Deep etched. Reduced one half in reproduction.

experimental evidence for the orientation dependence of the rate of growth in silicon ferrite.²⁵

The present investigation was undertaken in an attempt to clarify the difficulties and contradictions described above, and to rationalize both recrystallization textures and coarsening textures from the point of view of the "oriented growth" theory.

Experimental Procedure

The plan of the experiments was basically similar to that followed by Burgers and Basart.⁵ However, high purity aluminum was used in order to eliminate the possible effect of impurities on the texture relationships. Recent work indicated¹⁰ that it is possible to induce texture-dependent

coarsening in high purity aluminum. Table 1 gives the lot analysis of the aluminum.

Table 1 . . . Lot Analysis of High Purity Aluminum

	Per Cent
Si.....	0.002
Fe.....	0.002
Cu.....	0.002
Mg.....	0.003
Mn.....	0.001

The method of preparing the ingot was the same as previously used.¹⁶ Large crystals were grown by annealing 0.390 to 0.535 in. thick specimens for 3–4 days at 650°C. Crystals I and II were cold rolled 88 pct, and 90 pct respectively. Crystal III was compressed 75 pct between repeatedly greased ground steel plates. The deformed crystals were carefully cut into several specimens by a jeweler's saw. One specimen of each crystal was used for determining the deformation texture. The other specimens were annealed in salt baths at various temperatures and for various lengths of time.

Fig 1 gives the outlines of crystal I and its neighbors, after rolling, the location of the saw-cuts and specimen numbers. Spec. 7 was used for determining the deformation texture. As seen in Fig 2, crystal I had a large number of deformation bands extending parallel to the rolling direction. Spec. 4, (Fig 3), was annealed for 25 min. at 400°C, giving a uniformly fine recrystallized structure. Spec. 8, which had a common side with spec. 7, was annealed for 745 min. at 400°C, giving a partially coarsened structure, (Fig 4). This specimen was used to determine both the recrystallization texture and the orientation of several coarse grains. Since both structures were located in the same specimen, the relative orientation could be determined with a high degree of accuracy. For measuring the relative orientation of the deformed grain, spec. 7 was aligned by using its common edge with spec. 8. The specimens of crystals II and III were treated similarly.

The deformation texture, the re-

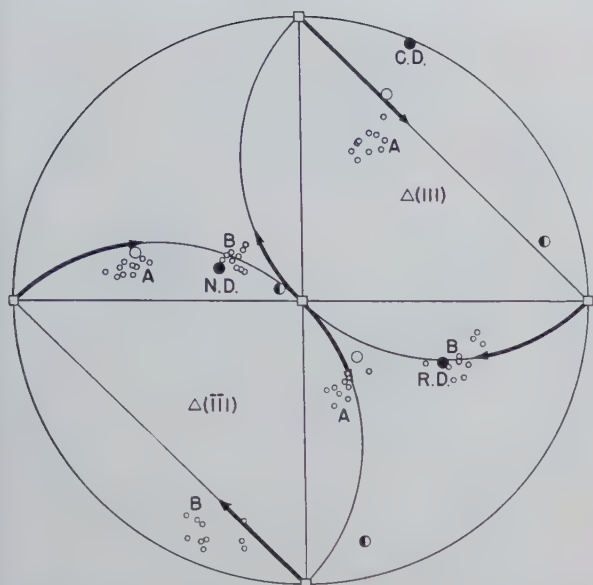


FIG 5—(100) pole figure for textures obtained in crystal I.

Recrystallization texture $\square$ is in standard position. The main component of the deformation texture $\circ$, and the deformation bands $\bullet$, as well as groups A and B of coarse grains $\circ$ are plotted in correct relative position, together with the rolling direction (R.D.), the cross direction (C.D.), and the normal direction (N.D.). Arrows correspond to 42° rotation around $[111]$ and 35° rotation around $[111]$ respectively.



FIG 6—(100) pole figure for textures obtained in crystal I.

Recrystallization texture $\square$, the two components of the deformation texture $\circ$ and $\bullet$, and group A coarse grains $\circ$ are plotted in such a way that the $[111]$ of the recrystallization texture is the center of projection. The rolling direction (R.D.), cross direction (C.D.), and normal direction (N.D.) are indicated.

crystallization texture and the coarsening texture were determined by means of an optical goniometer similar to that described by Barrett.¹⁷ The light source used was a 2 watt concentrated arc lamp, allowing a very high degree of collimation.

The annealed specimens were polished electrolytically and etched with the reagent described by Barrett.¹⁷ It was found that about 10 to 20 min. of etching were necessary to develop the cube planes accurately. In all measurements on single grains the measured angles between the cube planes approached 90° to an accuracy of better than 1 to 2°. It was found that the crystallographic definition of the etch pit faces in deformed crystals was improved when a somewhat modified etching reagent was used as given in Table 2.

Table 2 . . . Composition of Etching Reagent Used for Developing (100) Planes in Cold Worked High Purity Aluminum

Conc. HCl	80 cc
Conc. HNO ₃	25 cc
H ₂ O	20 cc
Conc. HF	5 cc

It was found that the texture of the fine grained recrystallized material is usually rather sharply defined, so that the mean orientation can be satisfactorily and easily determined by the optical method, if such a polycrystalline region is treated as if it was a single grain. Reproducible average orientations were obtained, and the angles

between the average cube planes so determined were close to 90°. The coarsening texture was determined by measuring the orientation of 15–20 large grains individually.

Results

Fig 5 gives the relationship between the deformation texture, the recrystallization texture and the coarsening texture obtained in crystal I. In order to show clearly the relative orientation of these textures, the pole figure is plotted for (100) poles in such a way that the recrystallization texture is in standard position. It is readily seen that the main portion of the deformed material as well as the deformation bands can be brought into fairly good alignment with the recrystallization texture by rotation around a $[111]$ direction. The coarse grains clearly fall into two groups (A and B), each with a well-developed preferred orientation. This fact is also noticeable in Fig 4, where one group of coarse grains appears black, and the other group gray. It is notable that the orientation of both groups of coarse grains deviates from that of the recrystallized matrix corresponding to a rotation around a $[111]$ direction. The angle of rotation from the deformation texture to the recrystallization texture is about 40°, that from the recrystallization texture to the group A coarse grains is of similar magnitude in the opposite direction

(or about 80° in the same direction), so that the orientation of group A coarse grains is fairly close to that of the original deformed single crystal. It is important to note that the group B coarse crystals have an entirely different orientation from either component of the deformed crystal, although they are also connected with the recrystallization texture by a $[111]$ rotation of about 35° (see Fig 1).

The extent of rotation around a $[111]$ direction can be best studied in the corresponding $[111]$ projection of the (100) poles. This is given in Fig 6 for the three successive generations of crystals: the deformation texture of crystal I (that of both the main portion and the deformation bands), the recrystallization texture and the group A coarse grains. It is apparent that with a fair degree of accuracy all these crystals have in common the $[111]$ direction which has been used as the center of projection. The group B coarse grains were omitted here, since they are connected with the recrystallization texture by rotation around a different $[111]$ direction. Similar pole figures were prepared for all cases investigated, in order to determine the angles of rotation.

Fig 7 shows the $[100]$ projection of the (100) poles for the recrystallization texture and for the coarse grains produced by the annealing of a specimen of the rolled crystal II for 25 min. at 400°C. In this instance it was found impossible to determine accurately the

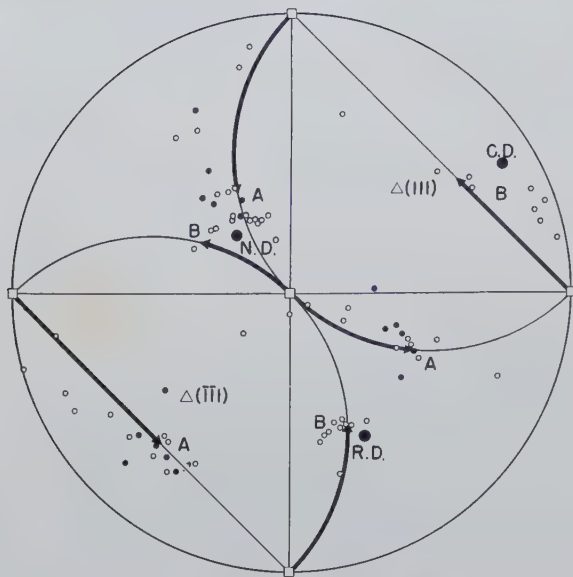


FIG 7—(100) pole figure for textures obtained in high purity aluminum crystal II.

Recrystallization texture $\square$ is in standard position. Groups A and B of coarse grains away from sawed edges $\circ$, and at sawed edges $\bullet$ are plotted together with rolling direction (R.D.), cross direction (C.D.), and normal direction (N.D.). Arrows correspond to 65° rotation around $[111]$ and to 50° rotation around $[111]$ respectively.

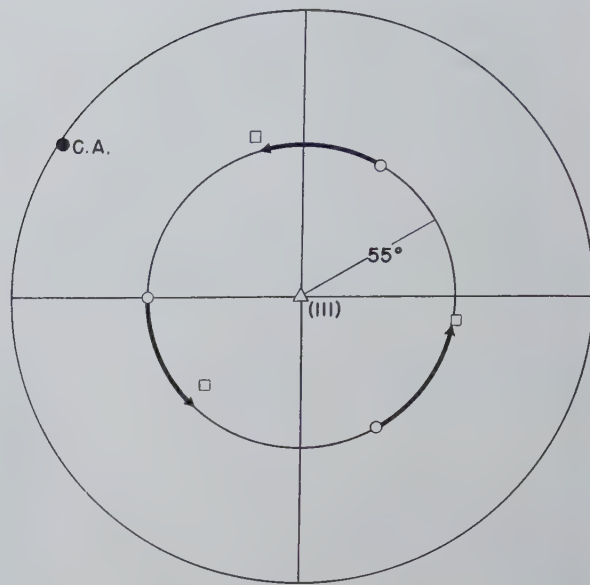


FIG 9—(100) pole figure for deformation texture $\circ$, and recrystallization texture $\square$ obtained in high purity aluminum crystal III.

(111) of the deformation texture is center of projection. The compression axis (C.A.) is indicated. Arrows indicate rotation of 47° .



FIG 8—Specimen of high purity aluminum originally consisting of two grains, rolled and annealed 12 sec. at 400°C , showing coarse grains at sawed edges.

Reduced one half in reproduction.

déformation texture by the optical method, as the deformation bands were small, numerous and inextricably interwoven. However, the recrystallization texture was fairly sharply defined. The coarse grains showed considerable scatter, but it is apparent that even this scattered orientation range is quite clearly related to rotations around one of two $[111]$ directions. Roughly, the scatter of one group of coarse crystals (A) may be expressed as a variation of the angle of rotation around $[111]$ from about 17 to 65° . The orientation of the other group of coarse crystals can be derived from that of the recrystallized grains by a rotation of approximately 50° around $[111]$.

It is of particular significance that in most specimens a tendency was noted for the coarsening to start near the two sawed edges. This is clearly shown in Fig 8. It was observed that considerable amounts of material had to be etched off the edge regions prior to annealing in order to eliminate this tendency. In Fig 7 the (100) poles for several of the grains growing from the edge are shown by solid circles. The orientation of most of these grains coincides well with the orientations of group A coarse grains, formed away from the edges.

The orientation of crystal III after compression was very similar to that shown in Fig 10, that is, the (110)

plane was close to the plane of compression. This compressed crystal had no deformation bands; its orientation was sharply defined. After annealing for 15 min. at 400°C it showed a sharply defined recrystallization texture. The relative orientation of the recrystallization texture and the deformation texture, which is shown in Fig 9 in a $[111]$ projection, corresponds to a rotation of about 47° around $[111]$.

Discussion of Results

The results indicate that the orientation of aluminum crystals grown in a highly oriented matrix, either by recrystallization (when the matrix is deformed) or by coarsening (when the matrix is recrystallized, fine grained), is related to the orientation of the matrix by means of a rotation around a $[111]$ direction. The extent of the rotation is about 30 to 50° in most cases as shown in Table 3.

According to Burgers and Louwerse,⁶ the orientation relationship between a compressed single crystal of commercial purity aluminum and the recrystallization texture corresponds to a rotation around a $[112]$ direction. Fig 10 shows a pole figure from their paper. In Fig 11 the same data are re-plotted in such a way that the $[100]$ poles of the deformed crystal are in standard position. It is apparent that the $[111]$ rotation expresses the orientation relationship between the compressed single

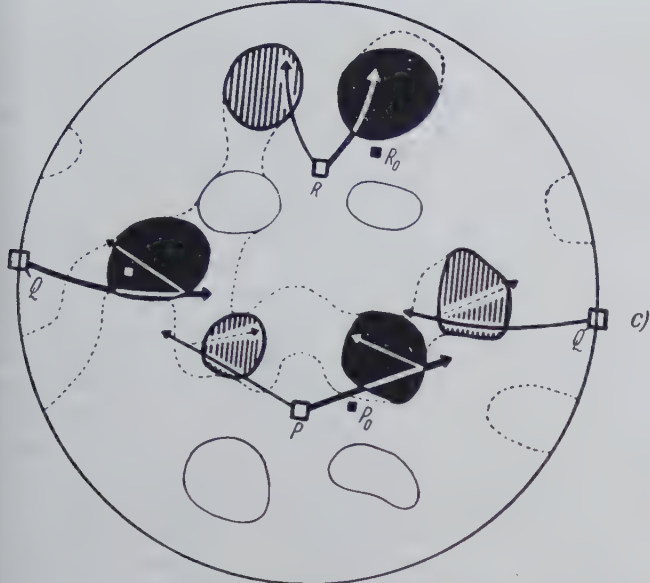


FIG 1 C—(100) pole figure for deformation texture $\square$ and recrystallization texture obtained by Burgers⁶ in a compressed aluminum crystal of commercial purity.

Black areas represent the main component (A), and shaded areas the other important component (B) of the recrystallization texture. Arrows indicate [112] rotations.

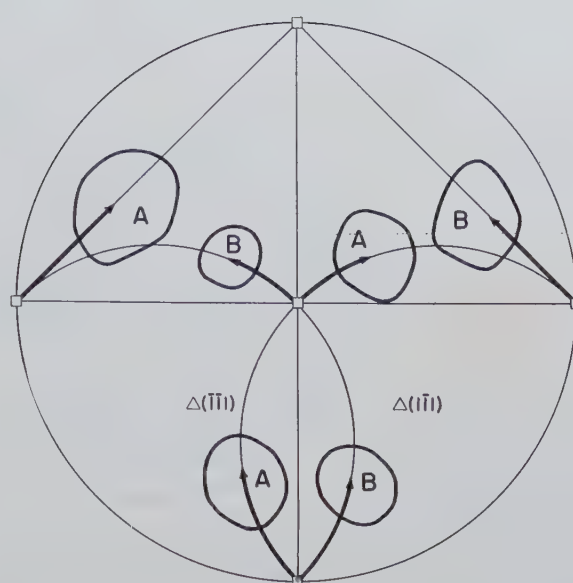


FIG 11—(100) pole figure for the data given in Fig 10, with the deformation texture in standard position.

Arrows indicate [111] rotations of 40° for A, and 37° for B.

crystal and either component of the recrystallization texture more accurately than the [112] rotations indicated by arrows in Fig 10. The orientation relationship between the deformation texture and the major component of the recrystallization texture is re-plotted in Fig 12 in a [111] projection, which again shows the excellent agreement of the data with the present interpretation. The agreement of the data reproduced in Fig 13 from ref. 6 (rolled single crystal of commercial aluminum) is again better with the [111] rotation than with the [112] rotation, as seen by comparing Fig 13 with Fig 14. It may be concluded, therefore, that the data presented by Burgers and his collaborators lend further support to the results described above. Furthermore, these data indicate that the impurities present in commercial aluminum do not affect the orientation relationship for recrystallization.

In all four instances where the relative orientation of deformation texture and recrystallization texture was determined (Fig 5, 9, 11 and 14), only one or two of the four possible [111] rotations were actually found to occur in noticeable amounts. It is to be noted that in the case of the two compressed crystals (Fig 9 and 11) the angle between the "active [111] axes and the compression axis is quite close to 90°. The [111] axes "active" in the recrystallization of the two rolled crystals (Fig 5 and 14) are those closest to the cross direction of rolling. (In the narrow deformation bands of crystal I the "active" [111]

Table 3 . . . Angle of Rotation Around [111] Direction in Degrees, Representing Orientation Change for Recrystallization and for Coarsening in a Highly Oriented Matrix

Material	Condition before last anneal	Source of information	Recrystallization	Coarsening
High purity aluminum	rolled single crystal	crystal I, this paper	40	42, 35
High purity aluminum	rolled single crystal	crystal II, this paper	47	17-65, 50
High purity aluminum	compressed single crystal	crystal III, this paper		
High purity aluminum	compressed single crystal	Barrett ⁸	45	
Commercial aluminum	compressed single crystal	Burgers and Louwerse, ⁶ as re-interpreted above	40, 37	
Commercial aluminum	rolled single crystal	Burgers and Louwerse, ⁶ as re-interpreted above	27, 33	
Tough pitch copper	rolled polycrystal	Bowles and Boas ¹⁵		30

direction was found to be about 2° further away from the cross direction than an inactive one. However, the accuracy of the measurements was not sufficient to make certain that this really represents an exception to the above criterion.) Since the direction of maximum compression in rolling is the normal direction, the criterion just described for rolled crystals is not unrelated to the one for compressed crystals.

The fact that the orientation of the recrystallized grains is derived from that of the deformed crystal by a [111] rotation implies, of course, that recrystallization nuclei of suitable orientation are available. It does not prove, however, that nuclei of other orientations do not exist. The occurrence of recrystallized grains of the above orientation range is favored probably because of their higher rate of growth in the deformed matrix. In the case of coarsening, this interpretation is

strongly supported by the orientation of the coarse grains nucleated at the sawed edge, as described above. Away from the edges the deformation texture is well-developed, and consequently the recrystallization texture is also quite sharply defined. Apparently, in a structure with such high degree of preferred orientation, there are but few grains with orientations deviating considerably from that of the mass, hence few "nuclei" for coarse grains. At the sawed edge the deformation is strongly heterogeneous. It may be assumed that at such locations tiny recrystallized grains of a large variety of orientations are formed upon annealing. Many of these may serve as "nuclei" in coarsening.* The fact that only those "nuclei" grow to large size which have the correct orientation with

* The described mechanism of "nucleation" of coarsening at points of inhomogeneous deformation may explain, at least in some cases, the phenomena of "forced secondary recrystallization" as reviewed by Burgers.²⁴

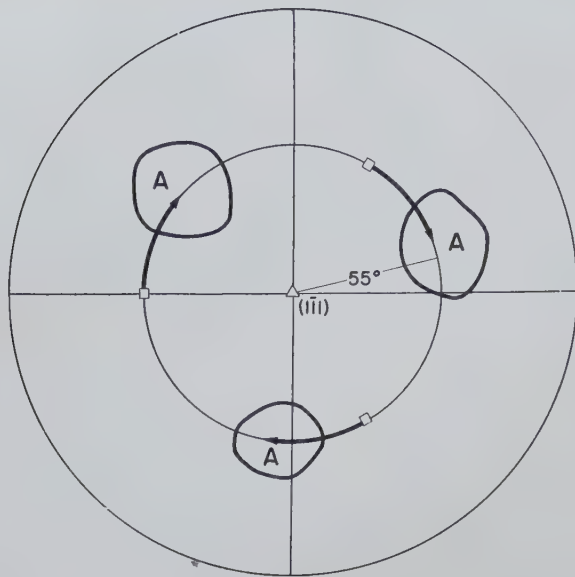


FIG 12—(100) pole figure for component A of the recrystallization texture, as given in Fig 10 and 11. Center of projection is (111) of the deformation texture.

respect to the highly oriented recrystallized matrix, indicates that the selective principle involved is the orientation dependence of the rate of growth. That the coarse grains are not "nucleated" by remnants of the deformation texture is shown by the group B coarse grains in Fig 5, the orientation of which is quite different from that of the deformation texture. However, the orientation of both group A and group B coarse grains can be accounted for on the basis of the "oriented growth" theory.

The analogous orientation selectivity

found for recrystallization and for coarsening in high purity aluminum shows that the same orientation dependence governs the rate of growth of the grains in both cases. There is no reason to assume, however, that this analogy implies essential identity of the two processes. In fact, present evidence tends to support the view that recrystallization is connected with the elastic bending of the lamellae of deformed crystals,¹⁸ whereas coarsening is connected with the surface energy of the grain boundaries.^{16,19}

It is interesting to recall that the

fiber texture of cold drawn polycrystalline aluminum is [111] in the fiber axis, and that the recrystallization texture, developed upon annealing, is the same.^{1,2,3} In the light of the present results, the retention of the [111] fiber texture in recrystallization is easily understood, even though each deformed grain changes its orientation upon recrystallization. Fig 15 shows schematically a [111] fiber texture. If each recrystallized grain deviates from its parent matrix only by a rotation around [111], which is also the fiber axis, its (100) poles will remain within the same ring-shaped area of the pole figure, and the resulting recrystallization texture will be the same as the deformation texture. Here, as in compressed single crystals, the "active" [111] axis (around which the re-orientation takes place) is perpendicular to the directions of maximum compression (the radial directions of the rod). In copper, too, the deformation fiber texture is mainly [111], and this is retained in recrystallization.²⁰ In view of this fact, and of the observations of Bowles and Boas on coarsening, the orientation relationships for copper appear to be very similar to those for aluminum.

Fig 16 shows the rolling texture for polycrystalline aluminum, according to v. Goeler and Sachs.⁴ If this is replotted with the cross direction in the center of the projection, as in Fig 17, it becomes apparent that, in a rough approximation, the major component of this texture may be considered as an

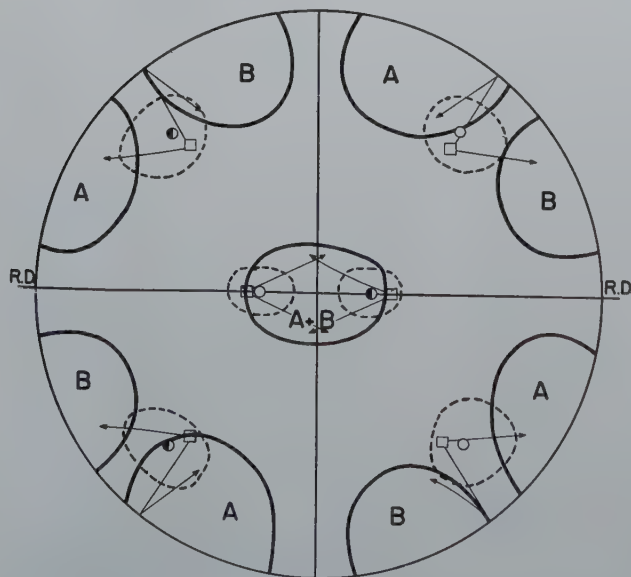


FIG 13—(100) pole figure for deformation texture (dotted lines), and for components A and B of recrystallization texture obtained by Burgers in rolled aluminum crystal of commercial purity.

Arrows indicate [112] rotations, as given by Burgers.

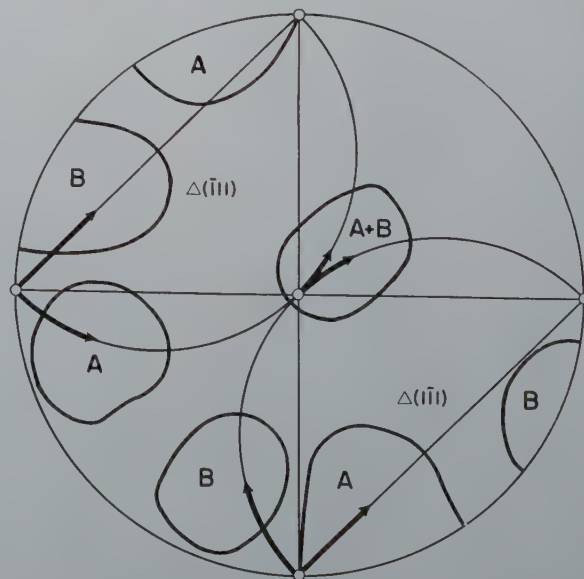


FIG 14—(100) pole figure for the data given in Fig 13, with one component of the deformation texture in standard position.

Arrows indicate [111] rotations of 27° for A, and 33° for B.

incomplete [111] fiber texture, with the cross direction as the fiber axis. Consequently, the often noted fact that the rolling texture of polycrystalline aluminum is fairly well retained upon recrystallization,⁴ may be visualized as described above for the fiber texture, even though the agreement cannot be expected to be as good as it is in the case of drawn aluminum. According to this interpretation, the "active" [111] axes in the various grains are all nearly parallel to the cross direction, in agreement with what was found above for rolled single crystals.

The behavior of rolled polycrystalline copper (with not more than about 80 pct reduction of area) is apparently very similar to that just described for aluminum. But it has been reported that the recrystallization texture of rolled silver²¹ and of certain face centered cubic copper alloys,²² such as brass, has mainly a (113) [112] position, even though the [111] fiber texture of cold drawn brass is apparently retained upon recrystallization.⁷ These conditions are not as yet clearly understood, and they are now being investigated at this laboratory.

It is known that certain body centered cubic metals have a [110] fiber texture, and that this is retained in recrystallization.²³ In analogy to the behavior of aluminum, it may be expected that in such metals the orientation relationship between a deformed grain and the recrystallized grains

growing in it may correspond to a rotation around a [110] direction. This expectation is also being tested at the present time.

Acknowledgments

The authors wish to express their appreciation to Mr. Philip R. Sperry for his conscientious and competent assistance with the experiments, and to the Aluminum Co. of America for

the grant of high purity aluminum. The continued interest and encouragement of Drs. A. R. Troiano, H. A. Dix, Jr., and A. J. Phillips is gratefully acknowledged. This work was supported by the office of Naval Research, U. S. Navy, Contract No. N6 ori-165, T.O. 1. The helpfulness of Messrs. I. R. Kramer, J. J. Harwood and James Bryson of the Materials and Mechanics Branch of the O.N.R. was of great value.

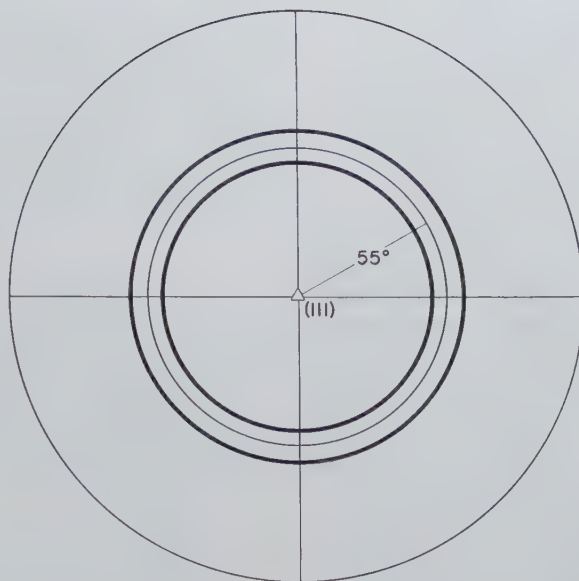


FIG 15—Schematic (100) pole figure for [111] fiber texture, with fiber axis as the center of projection.

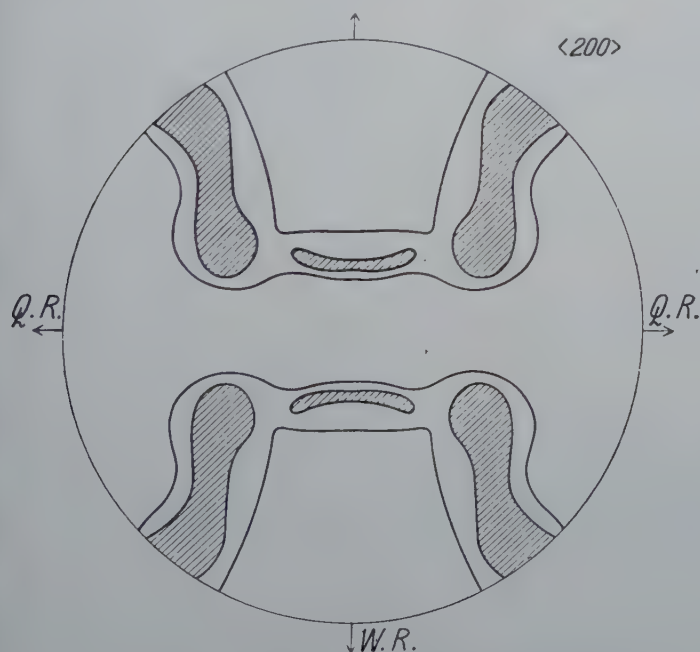


FIG 16—(100) pole figure for deformation texture of rolled polycrystalline aluminum, according to v. Goeler and Sachs.⁴ Plane of projection is the rolling plane. Rolling direction indicated by W. R.

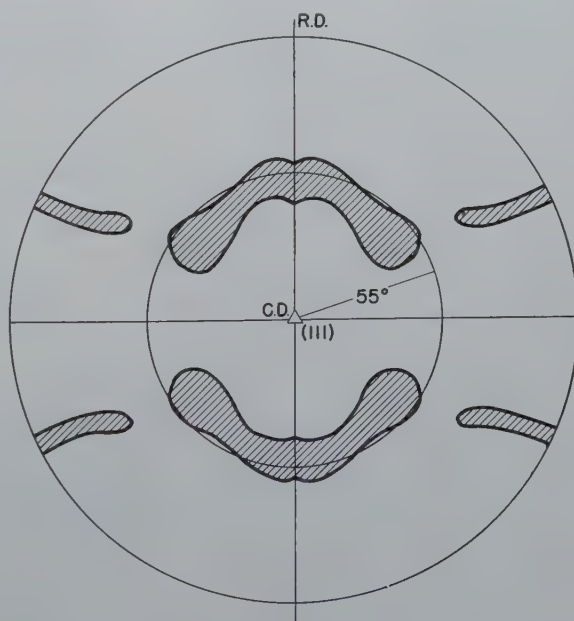


FIG 17—(100) pole figure for the data given in Fig 16, with the cross direction as the center of projection. The rolling direction is indicated.

Conclusions

1. The orientation of the recrystallized grains growing in a rolled or compressed aluminum single crystal is related to the main orientation of the deformed crystal by rotation around a [111] direction. The extent of rotation varies between about 30 and 50°. The orientation relationship for recrystallization does not appear to be affected by impurities present in commercial aluminum.

2. Since the axis of the rotation, which may be used to describe the orientation relationship between deformation texture and recrystallization texture for aluminum single crystals, is identical with the axis of the fiber texture of cold drawn polycrystalline aluminum, the retention of the [111] fiber texture in the recrystallization of this material is explained.

3. The orientation of the coarse grains growing in a matrix of fine grained recrystallized high purity aluminum with sharply defined texture is related to the main orientation of the matrix by rotation around a [111] direction. The extent of rotation may vary over wide limits, but it is usually about 30 to 50°. Important components of the coarsening texture may strongly deviate from the deformation texture. Consequently there is no reason to assume that the coarse grains are "nucleated" by remnants of the deformed material.

4. The orientation of coarse grains growing in a matrix of fine grained recrystallized high purity aluminum with sharply defined texture, from points where recrystallized grains with a large

variety of orientations are available (sawed edges) is the same as that of the coarse grains formed elsewhere. This indicates that the coarsening texture is determined by the orientation dependence of the rate of growth, rather than by "oriented nucleation."

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Correction June Issue

"The Statistical Nature of the Endurance Limit." By J. T. Ransom and R. F. Mehl. On page 365, June 1949 *Journal of Metals Transactions*, caption for Fig 2 should read, "Scatter Range for 210 Specimens."

Annealing Twins in Copper and 70-30 Alpha Brass*

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Cyril Stanley Smith in his classic 1948 Institute of Metals Lecture¹ noted in the case of annealing twins that: "It is well known that approximately the same number of twins per grain and the same general geometry are maintained in large grains as in small ones, a fact that certainly suggests a mechanism such as the above where boundaries and twins grow proportionately together. If this view is correct, a spherical grain should have no mechanism for keeping its twins." Such a constancy of twins per grain would be of particular interest as a tool for the study of the origin of annealing twins. It has been proposed by Mathewson² and later by Barrett³ that in face-centered cubic metals slip occurs in the {111} plane by <112> shears which combine in pairs to produce the macroscopically observed <110> movement. This proposal is schematically inviting since <112> slip permits the atoms of one plane to move along the interatomic valleys of its neighbor rather than up the hills across the atom sites. There is strong experimental evidence⁴ that annealing twins result from the growth of nuclei in the form of "twin faults" produced by single <112> shears.

It is possible that more twin faults would form at low deformation temperatures than at high ones, because the more rigid lattice may increase the tendency to "freeze" the twin faults

and prevent their dissipation in slip. This is suggested by (1) the greater resistance to slip at low temperatures and (2) greater tendency toward mechanical type twinning at low temperatures (a summary of these factors is presented by Seigle and Brick⁵). These considerations would suggest the following:

- Constant twin/grain ratio for specimens deformed constant amounts at a constant temperature, but annealed at various temperatures above the recrystallization temperature.
- Larger twin/grain ratios for recrystallized specimens previously deformed constant amounts at lower temperatures.

The purpose of this investigation is to test these considerations.

Cleveland Meeting, October 1949.
TP 2677 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received May 2, 1949.

* The data in this paper were taken from reports submitted by Y. C. Liu and S. F. Reiter to the School of Engineering, Yale University, in candidacy for the Degree of Master of Engineering, May 1949.

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¹ References are at the end of the paper.

Experimental Procedure and Results

One half in. rods of phosphorous deoxidized electrolytic copper and 70-30 alpha brass (68.36 pct Cu, 0.005 pct Pb, 0.01 pct Fe, 31.62 pct Zn remainder) were rolled to a thickness of 0.350 in., milled to specimens $0.350 \times 0.437 \times 2$ in., and annealed at 700°C to an average grain size of 0.065 and 0.090 mm, respectively. These specimens of each metal were then rolled 50 pct reduction in thickness and annealed one hour under conditions which were all combinations of the following:

Rolling Temperatures, °C	Annealing Temperatures, °C
-70	400
25	500
200	600

Standard metallographic techniques⁶ were used to prepare specimens. By the method of lineal analysis⁷ using a minimum of ten samples of fifty grains each for each specimen, the ratios of twins* per total grains, twins per twinned grain, twin families* per total grains and twins per twin family were determined. Recrystallized grain size was determined by A.S.T.M. comparison methods.⁸ All observations were made on the rolling plane.

The results are tabulated in Table 1.

* Twins were counted as the number of distinguishable twins on the polished surface even though many of them were parallel. Twin families were counted as the number of different twin directions in a grain, a maximum of four.

Table 1 . . . Annealing Twins in Rolled and Recrystallized Copper and Alpha Brass

Rolling Temperature.....	-70°C			25°C			200°C		
Annealing Temperature.....	400°C	500°C	600°C	400°C	500°C	600°C	400°C	500°C	600°C
Copper									
Twins per total grains.....	0.95 ± 0.04	0.95 ± 0.04	1.08 ± 0.05	1.04 ± 0.08	1.14 ± 0.05	1.11 ± 0.05	1.11 ± 0.07	1.14 ± 0.07	1.06 ± 0.05
Twins per twinned grains.....	1.65 ± 0.05	1.67 ± 0.05	1.58 ± 0.05	1.78 ± 0.09	1.69 ± 0.06	1.68 ± 0.03	1.82 ± 0.08	1.82 ± 0.07	1.65 ± 0.09
Twin families per total grains.....	0.70 ± 0.01	0.65 ± 0.02	0.76 ± 0.03	0.68 ± 0.04	0.75 ± 0.02	0.78 ± 0.02	0.70 ± 0.03	0.71 ± 0.03	0.73 ± 0.02
Twins per family.....	1.35 ± 0.05	1.46 ± 0.04	1.41 ± 0.03	1.55 ± 0.06	1.52 ± 0.04	1.43 ± 0.03	1.56 ± 0.05	1.62 ± 0.05	1.46 ± 0.05
Grain Size, mm.....	0.025	0.035	0.045	0.035	0.035	0.045	0.045	0.045	0.045
Brass									
Twins per total grains.....	6.30 ± 0.28	5.85 ± 0.25	2.03 ± 0.08	6.17 ± 0.26	7.20 ± 0.23	1.87 ± 0.07	6.88 ± 0.23	7.56 ± 0.37	1.90 ± 0.07
Twins per twinned grains.....	6.70 ± 0.26	6.25 ± 0.20	2.23 ± 0.07	6.86 ± 0.25	7.61 ± 0.23	2.08 ± 0.08	7.58 ± 0.24	7.90 ± 0.40	2.16 ± 0.06
Twin families per total grains.....	1.83 ± 0.04	1.84 ± 0.05	1.24 ± 0.03	1.74 ± 0.03	2.01 ± 0.03	1.21 ± 0.03	1.83 ± 0.03	1.99 ± 0.04	1.26 ± 0.05
Twins per family.....	2.44 ± 0.11	3.16 ± 0.10	1.64 ± 0.03	3.51 ± 0.08	3.59 ± 0.10	1.52 ± 0.04	3.79 ± 0.11	3.79 ± 0.19	1.51 ± 0.03
Grain Size, mm.....	0.015	0.025	0.090	0.025	0.025	0.090	0.025	0.035	0.090

Discussion of Results

From Table 1 the following observations are apparent:

1. Under similar conditions of rolling and annealing, more annealing twins are found in 70-30 alpha brass than in copper, both from the standpoint of total number of twins and number of twin families.

2. In the case of copper, the various twin ratios are essentially independent of the annealing temperature as suggested by Smith.¹

3. In the case of 70-30 alpha brass, significantly fewer twins were formed at the highest annealing temperature under all rolling conditions. However, it was noted that a large number of nearly spherical grains survived at this temperature, again in accordance with the suggestions of Smith.¹

The tendency toward forming annealing twins is slightly but probably not significantly less for lower temperatures of deformation.

4. In the case of copper, by comparing twins per total grains with twins per twinned grains, it appears that after the 600°C anneal there were about the same number of twins per grain but significantly more twin-free grains than after the 400 and 500°C anneals. In the case of brass there were significantly fewer twins per grain but about the same number of twin-free grains at 600°C.

Observation (1) suggests that twin

faults form more readily in 70-30 alpha brass than in copper. Thus the tendency to form annealing twins is a direct function of the elastic anisotropy of the metal (that is, brass is more anisotropic than copper). This can be extrapolated to aluminum, which is nearly isotropic and in which annealing twins possibly have not been observed. It suggests that twin faults form more readily if the elastic modulus normal to the slip plane is significantly larger than that parallel to it.

Observations (2) and (4) suggest that in copper there is a tendency for twin-free grains to grow at the expense of grains containing twins, particularly those with few twins. Observations (3) and (4) suggest that in alpha brass many twins form at lower annealing temperatures (as reported by Maddigan and Blank,⁹ but that the twins coalesce within the grains at higher temperatures (600°C). This coalescence apparently starts when the grains have grown sufficiently to approach a spherical shape.

While more twin faults may have formed at lower deformation temperatures, in these experiments a proportionately larger number of them did not grow to annealing twins. Possibly the increased activity of the atoms at the annealing temperature permitted most of the twin faults to be absorbed.

Summary

70-30 alpha brass has greater tend-

encies toward annealing twin formation than copper. The twins per grain ratio is essentially constant for copper and 70-30 alpha brass over a wide range of deformation temperatures and annealing temperatures, except for the alpha brass at 600°C where an appreciable reduction in this ratio was found.

Acknowledgment

The interest, encouragement and counsel of Professor C. H. Mathewson are gratefully acknowledged. The phosphorous deoxidized electrolytic copper was furnished by the Scovill Manufacturing Co., Waterbury, Conn., and the 70-30 alpha brass rod by the American Brass Co., Torrington, Conn. Dr. Robert Maddin read and criticized the manuscript.

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The Yielding and Strain-aging of Carburized and Nitrided Single Crystals of Iron

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Introduction

Annealed, polycrystalline, low carbon steel exhibits a phenomenon known as the "yield point." If such a steel is loaded in tension, the load increases steadily with elastic strain, drops suddenly, fluctuates about some constant load value for a time and then rises with further strain. The load at which the sudden drop occurs is called the "upper yield point"; the load at which deformation proceeds at constant load is known as the "lower yield point"; the amount of elongation at constant load is termed the "yield point elongation."

Deformation which occurs during the yield point elongation is heterogeneous in nature, deformed metal existing next to undeformed metal. The drop in load from the upper yield point is concurrent with the formation of a band of deformed metal which usually appears at a stress concentration such as a fillet and at an angle of approximately 45° with the direction of pull. As deformation proceeds during the yield point elongation, the deformed region grows until the entire specimen has been strained an amount equal to the strain in the first band.

A phenomenon very often associated with the yield point is that of strain aging. Aging in steels may be of two types: quench aging, or precipitation hardening, and strain-aging. Strain aging differs from precipitation hardening in that if the metal is merely strained instead of given a solution anneal and quenched, it then undergoes changes in properties similar to those

observed in quench aging.

The yield point effect and strain aging are most markedly exhibited by low carbon steel, but have been observed in other alloys. Numerous explanations for the mechanisms of both phenomena have been advanced, but the evidence for the acceptance of any of the hypotheses is incomplete. One of the explanations of the yield point phenomenon frequently proposed is that there exists a honeycomb of grain boundary material that supports the load above the yield strength of the ferrite and that when this network ruptures, the ferrite flows with no increase in load. This notion was first advanced by Dalby¹ in 1913 and has been since put forward in more concrete form by many investigators. The testing of this hypothesis was, in part, the object of the present investigation. Similarly, the mechanism by which

strain aging occurs remains to be explained. Presumably, it is a precipitation hardening phenomenon similar to quench aging, the precipitation being initiated by strain. However, it is not known whether the process is a solution and precipitation effect or a change in solubility due to strain.

One obvious method of determining whether or not the yield point is a grain boundary phenomenon is to determine if single crystals exhibit a yield point. Low and Gensamer² have shown that carbon and nitrogen are responsible for the yield point and aging in steel and that these effects can be eliminated by the removal of these elements through wet hydrogen purification. Single crystals that have been grown in wet hydrogen treated material must then be recarburized or re-nitrided in order to evaluate the effect of grain boundaries. In the present investigation, single crystals of iron which had been annealed in wet hydrogen to remove the carbon and nitrogen were cut into two specimens having the same orientation with respect to the tension axis. Of these, one specimen was pulled in tension in the wet hydrogen treated state and the other was carburized or nitrided and tested similarly. All the crystals were examined for a yield-point and strain aging.

The grain boundary hypothesis for the explanation of the yield point in steel has been tested only indirectly previously. Arrowsmith,³ Andrew and Lee,⁴ Edwards and Pfeil,⁵ Fell,⁶ Ludwik and Scheu,⁷ Winlock and Leiter⁸ and others found a decrease in proportional limit and yield point effect with an in-

Cleveland Meeting, October 1949.
TP 2603 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received January 31, 1949.

This paper is based upon a thesis submitted by Harry Schwartzbart to the Pennsylvania State College in partial fulfillment of the requirements for the degree of M.S. in metallurgy.

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¹ References are at the end of the paper.

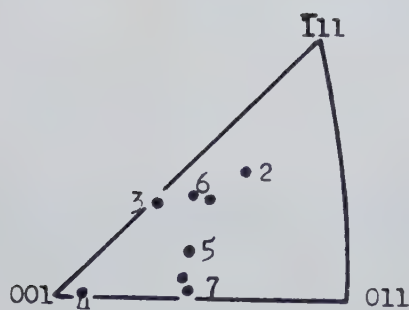


FIG 1—Orientations of the single crystals.
Checks were made on Crystals 6 and 7.

crease in grain size. Edwards and Pfeil⁵ did not find a yield point in a single crystal of hydrogen purified mild steel, but the carbon and nitrogen were probably absent in their crystals. Single crystals of iron grown in wet hydrogen purified mild steel were tested under dead weight loading by Gensamer and Mehl⁹ who found that at stresses at which yielding occurs, the rate of elongation is initially low, then increases, and finally decreases to zero. They point out that this behavior will

be observed in material whose flow curve shows a drop in load at yielding. Gensamer¹⁰ tested similar crystals in a conventional tension testing machine; these crystals showed no drop in load but the initial plastic elongation took place at substantially constant load. These crystals had not had all of the carbon and/or nitrogen removed. Holden and Hollomon¹¹ in an investigation contemporaneous with the present one found no yield point in carburized or nitrided single crystals of iron. A point-by-point comparison of the experimental procedures used in the two investigations reveals the following principal differences:

1. Holden and Hollomon's crystals were made from aluminum killed stock while those of the present investigation were made from silicon killed material. The presence of an excess of aluminum might conceivably affect the behavior of nitrided single crystals by combining with the nitrogen but would not be expected to have a similar effect in the case of carburized crystals:

2. Holden and Hollomon used $\frac{1}{8}$ -in. ga. length SR-4 strain gauges for measuring strain while the present authors used 1-in. Tuckerman gauges. The shorter gauge length used by Holden and Hollomon would tend to obscure the "drop in load" feature of the yield point, but the "strain at constant load" portion should have been observed if present.

3. Holden and Hollomon's single crystals were slowly cooled following carburizing and nitriding, whereas the present authors' were air cooled. There is no apparent reason why this difference in cooling rate should lead to the reported differences in yielding behavior.

Up to the present time, no satisfactory explanation has been found for the differing results of the two investigations.

Experimental Procedure

The single crystals used in this investigation were prepared at Carnegie Institute of Technology by the strain-anneal method and are similar to the crystals which were used in the investigation of Gensamer and Mehl⁹ discussed earlier. Details of their fabrication can be obtained from their paper. The bar stock from which the crystals were grown contained 0.36 pct Mn, 0.010 pct P, 0.025 pct S and 0.09 pct Si and it is to be expected that the single crystals contained substantially the same amounts of these elements.

Six crystals were used in the present investigation, all of which were initially in the form of $\frac{3}{8}$ in. diam round bars. No X ray examination for crystal perfection was made; however, macroscopic examination after etching to develop etch-pits did not reveal any occluded crystallites or evidence of gross crystal imperfections.

The crystals were found to contain 0.003 pct carbon when analyzed by the low pressure vacuum fusion method; hence it was not known if they had been adequately decarburized to eliminate the yield point and strain aging since this amount of carbon lies near the limit found by Low and Gensamer for these two characteristics in polycrystalline specimens. Therefore, further wet hydrogen treatment was performed on the crystals after machining into tensile specimens for this investigation.

The orientations of all the crystals were determined and the orientations

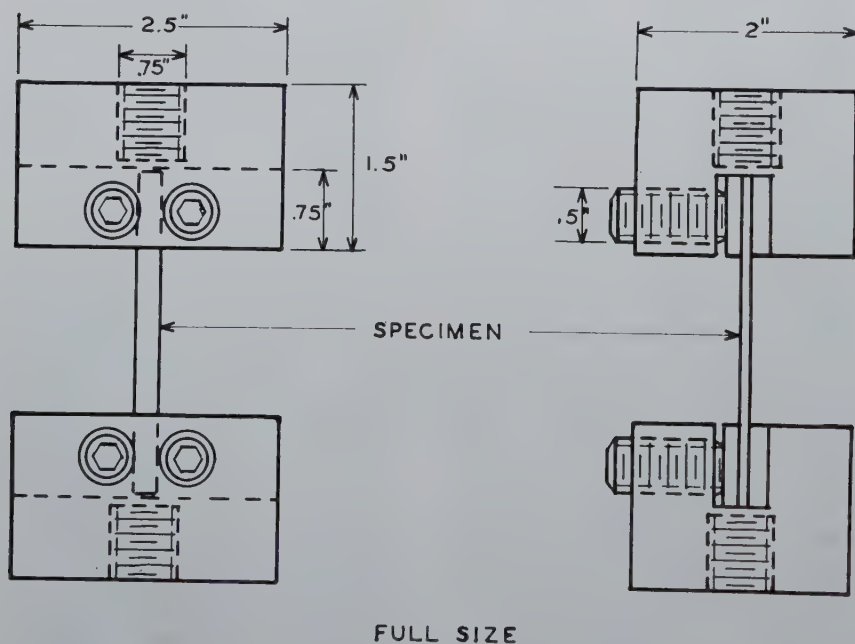


FIG 2—Grips used in single crystal tension tests.

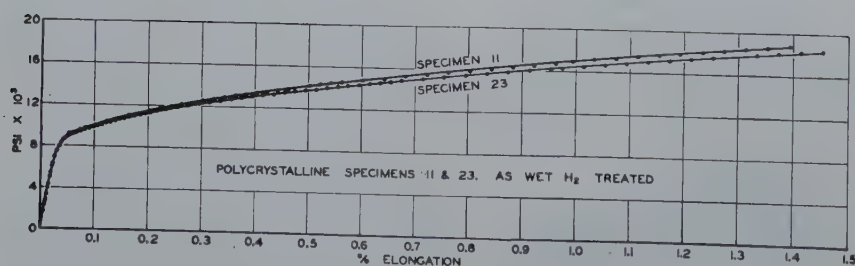


FIG 3—Stress-strain curves for polycrystalline specimens 11 and 23.
As wet hydrogen treated.

of the tension specimen axes are plotted in the stereographic triangle of Fig 1. The method used to determine orientations was similar to the etch pit technique described by Barrett and Levenson.¹² Since the success of this investigation depended on the fact that the specimens being compared were cut from the same single crystal and, therefore, had the same orientation it was not essential that the orientation be determined with a high degree of accuracy.

Each of the single crystal bars was sawed in half longitudinally and two halves ground flat to give two specimens having the same orientation with respect to the tension axis. The specimens consisted of simple rectangular prisms with no reduced section. The dimensions of the specimens before testing varied from 0.1505 in. to 0.3036 in. wide by 0.0416 in. to 0.0578 in. thick. The length of the specimens between the grips varied from $1\frac{5}{16}$ in. to approximately $2\frac{1}{2}$ in.; the distance between grips was the same for all specimens from a single crystal. All of the specimens were composed of a single crystal between the grips except the specimens of Crystal 2 which had a grain boundary about $\frac{3}{4}$ in. from one grip. In this case the knife edges of the extensometer did not contain the grain boundary between them but were placed entirely on one crystal at least $\frac{1}{4}$ in. away from the grain boundary. The results obtained on Crystal 2 are consistent with those for the other crystals.

After machining, the specimens were heated to 720°C in tank hydrogen, then held for the amounts of time shown in Table 1 in an atmosphere of hydrogen containing water vapor in the percentages noted in the same table, and air cooled. The flow rate of gas through the furnace was 2 cfhr which is equivalent to a linear flow rate of 0.037 fps. The specimens after cooling in air were coated with a thin oxide film; this was removed in 10 pct HNO₃. In some cases there was a layer of fine crystals on the specimens caused by recrystallization of the machined (cold worked) surfaces. These fine crystals were removed by alternately etching in 10 pct HNO₃ and grinding, using extremely light passes.

It can be seen in Table 1 that not all the crystals received identical wet hydrogen treatments. Since it was observed in the cases of Crystals 2, 3 and 4 that a slight yield point indication was obtained on the uncarburized

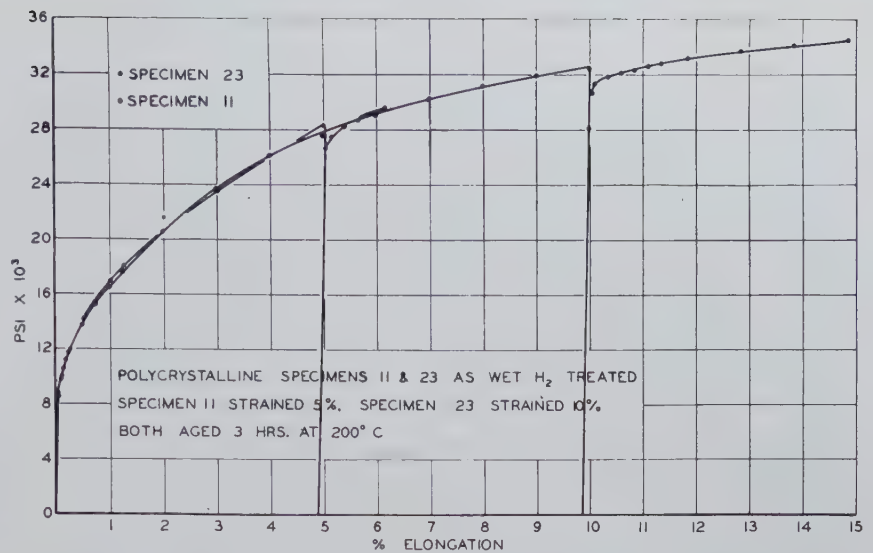


FIG 4—Composite stress-strain curves for polycrystalline specimens 11 and 23. As wet hydrogen treated.

Table 1 . . . Wet Hydrogen Treatments of Single Crystals

Temp.—720°C; linear flow rate—0.037 fps

Crystal No.	Time of Treatment, Hr	Vol. Pct H ₂ O
2, 3, and 4	6¾ 8½	10 (saturator at 46°C) 3 (saturator at room temperature)
5 and 7	8½ + 11¾	3 (saturator at room temperature) 10 (saturator at 46°C)

specimen in the test after aging it was desirable to learn if longer treatments would reduce the carbon and/or nitrogen to the level of complete elimination of yield point indications either before

or after aging. The treatment in which the saturator was at room temperature was a mistake. The furnaces used for all the heat treating were of the nichrome wound electric resistance type with a Sillimanite tube as the heating chamber.

One of the specimens cut from Crystals 2, 3, and 4 was heated to 700°C in tank hydrogen, then held for 10 min. in an atmosphere of hydrogen saturated with normal heptane at 22°C (this will yield a mixture containing about 5 pct heptane), and air cooled. These conditions and short carburizing time were chosen to prevent the introduction of a second phase in the crys-

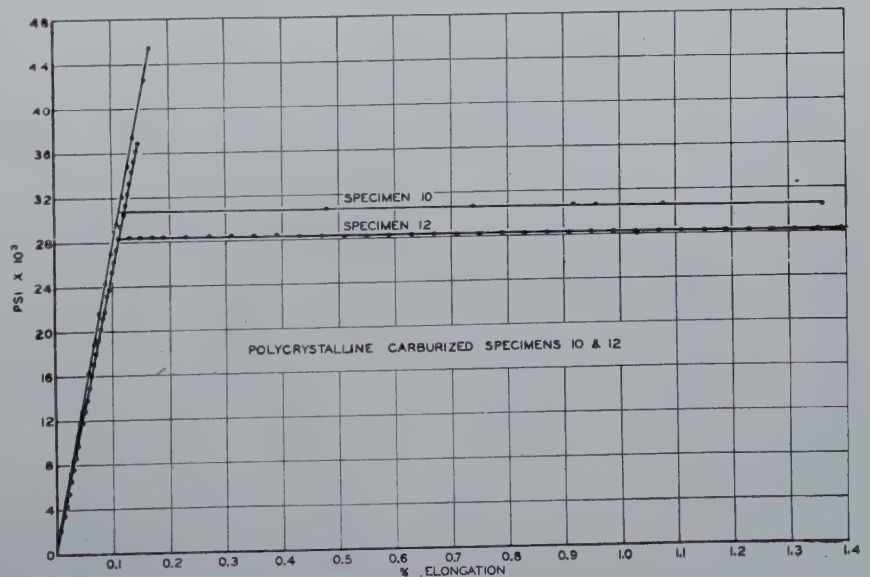


FIG 5—Stress-strain curves for polycrystalline carburized specimens 10 and 12.

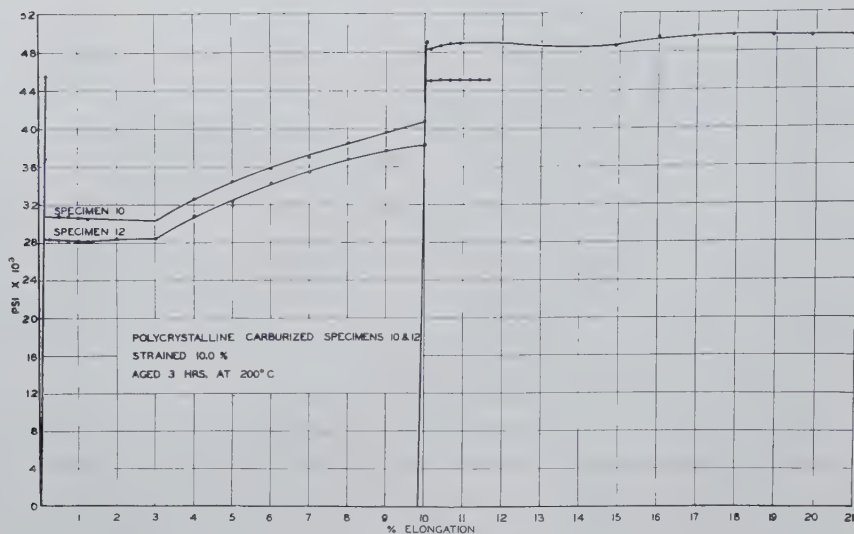


FIG 6—Composite stress-strain curves for polycrystalline carburized specimens 10 and 12.

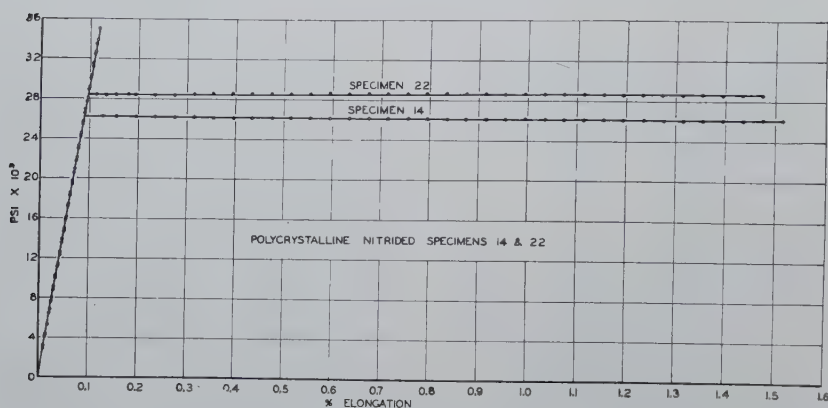


FIG 7—Stress-strain curves for polycrystalline nitrided specimens 14 and 22.

Table 2 . . . Mechanical Properties and Chemical Analyses of Carburized, Nitrided and As-hydrogen-purified Polycrystalline and Single Crystal Specimens

Crystal or Specimen Number	Condition	Yield Strength, Psi $\times 10^3$	Stress at 4 Pct Strain, Psi $\times 10^2$	Pct Which Carburization or Nitriding Raised the Stress-strain Curve at 4 Pct Strain	Pct Strain Aging	Pct Carbon (Duplicate Analyses)		Pct Nitrogen
Single Crystal 2	Uncarburized	10.0	13.0		0	0.002	0.002	
Single Crystal 3	Carburized	12.0	15.0	15	10	0.005	0.005	
Single Crystal 4	Uncarburized	10.5	13.5		0	0.002	0.002	
Single Crystal 5	Carburized	11.5	14.0	4	7	0.005	0.005	
Single Crystal 6*	Uncarburized	10.0	14.0		0			
Single Crystal 7	Carburized	12.5	15.0	7	13			
Single Crystal 8	Not nitrided	8.5	11.0		4			0.0004
Single Crystal 9	Nitrided	12.0	14.0	27	17			0.0070
Single Crystal 10	Not nitrided	16.0	17.0		9			
Single Crystal 11	Nitrided	18.0	19.5	15	18			
Single Crystal 12	Not nitrided	12.5	15.5		0			0.0004
Single Crystal 13	Nitrided	14.0	16.5	6	24			0.0070
Polycrystalline Specimen 11	As wet H ₂ treated	9.0	26.0		0	0.002	0.002	
Polycrystalline Specimen 23	As wet H ₂ treated	9.0	26.0		0			0.0007
Polycrystalline Specimen 10	Carburized	29.5	32.5	25	17			
Polycrystalline Specimen 12	Carburized	28.0	31.0	19	17	0.010	0.011	
Polycrystalline Specimen 22	Nitrided	28.5	33.5	29	17			0.0140
Polycrystalline Specimen 14	Nitrided	26.0	30.5	17	18			

* The specimens cut from this crystal were given insufficient wet hydrogen purification.

tals. The flow rate of gas through the furnace was 1.5 cfhr or 0.028 fps.

To make certain the carburization was effective, control specimens of wet hydrogen purified polycrystalline low carbon rimmed steel (ASTM grain size 8) were treated with the single crystals. The control specimens had been cut from 0.036 in. sheets previously wet hydrogen purified at 720°C for 4 hr and known to be free of either a yield point or aging. Specimens adjacent to the control specimens in the original wet hydrogen treated sheet were tested in tension and examined for yield point and strain aging. From the stress-strain curves reproduced in Fig 3, 4, and 5, it is evident that the control specimens had received sufficient purification to eliminate the yield point and strain aging and that if they exhibited these effects after carburization they were due to that carburization. The polycrystalline specimens were standard ASTM sheet specimens. Air cooling after carburization produced a scale on the specimens which was removed with 10 pct HNO₃. No recrystallization, due to carburizing, could be observed on the etched specimens.

The train used for nitriding was as follows: Tank hydrogen → CaCl₂ → empty Erlenmeyer flask at liquid air temperature → activated charcoal at liquid air temperature → liquid NH₃ saturator at -77°C → nitriding furnace. The CaCl₂ and liquid air trap were driers; the activated charcoal at -190°C was necessary for the removal of hydrocarbons present in tank hydrogen;² the ammonia saturator was maintained at -77°C to keep the concentration of ammonia in the hydrogen low enough (6 pct) to prevent the formation of iron nitrides.¹³ A venturi type flow meter between the hydrogen tank and the CaCl₂ drier indicated the flow rate which was maintained at 1.5 cfhr or 0.127 fps. The nitriding procedure was as follows: The single crystal specimens and, as in the case of carburization, the polycrystalline control specimens were placed in the cold furnace; dried ammonia vapor was introduced into the saturator where enough was condensed for one run; the specimens were heated to 500°C in hydrogen passing through the above train but by-passing the ammonia saturator; once at temperature the saturator was cut into the system by a suitable arrangement of glass T's and pinchcocks. Nitriding proceeded for 3 hr at 500°C after which the specimens were air cooled. This produced a light scale

which was removed in 10 pct HNO_3 . No recrystallization, due to nitriding, could be observed on the etched specimens.

Tensile tests were made on a Baldwin-Southwark 60,000 lb hydraulic machine with an Emery weighing system. Load readings were taken on the 1200 lb range dial of the machine to the nearest pound. The rate of head movement on all tests was approximately 0.0022 in. per min. Elongations up to about 1.6 pct were measured with a Tuckerman optical extensometer on a 1 in. ga length with a precision of 2×10^{-6} in. per in. Light punch marks were placed 1 in. apart on the single crystals and 2 in. apart on the polycrystalline specimens; elongations greater than 1.6 pct were then measured to the nearest 0.01 in., using dividers.

Axiality of loading was achieved through the use of modified Robertson shackles wherein the load is applied at the point of contact between a hardened steel plane and a steel ball. That part of the shackle which contained the threaded end of the specimen, adapter or grip could be shifted in relation to the stress axis by means of positioning screws thus correcting for any eccentricity in the system. A test for axiality is the requirement that upon a change in load in the elastic range all four sides of the specimen elongate or contract the same amount. In this investigation the specimens were aligned to the extent that, upon a change in load in the elastic range, the changes in length of any two opposite sides were within 6 pct of each other.

The polycrystalline specimens were gripped in Templin sheet grips. A special set of grips was used for the single crystals; these are illustrated in Fig 2. The specimen was fastened in the grips with the latter out of the tensile machine; the complete assembly as shown in Fig 2 was then transferred to the testing machine, with great care being exercised to avoid bending of the specimen.

The single crystals before testing had all been etched in 10 pct HNO_3 . The only difference in treatment of the two specimens cut from any single crystal was the carburization or nitriding treatment. All crystals were strained 5.0 pct, the load removed and the specimen (still in the grip assembly) was placed immediately in an oven at 200°C for 3 hr after which they were pulled again and examined for strain

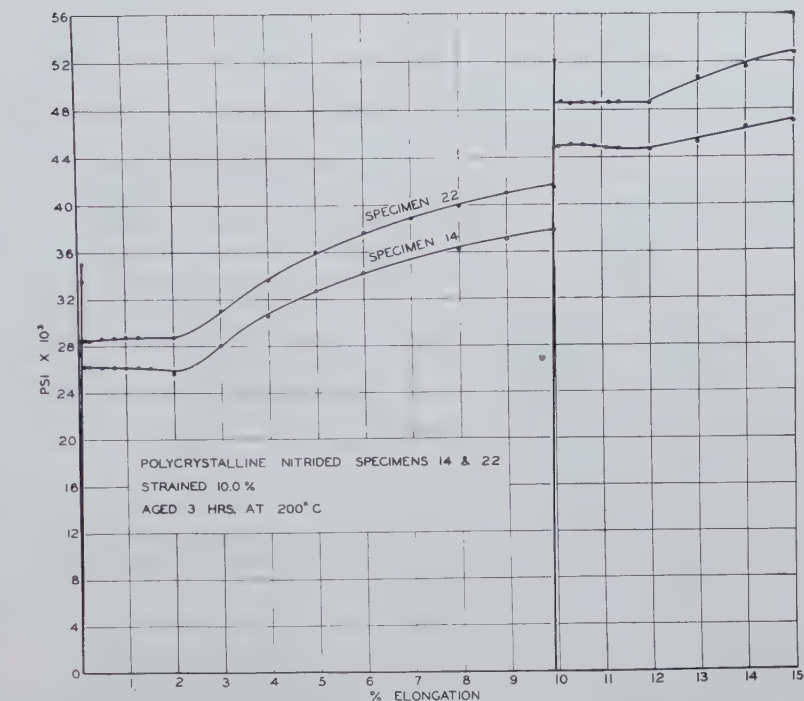


FIG 8—Composite stress-strain curves for polycrystalline nitrided specimens 14 and 22.

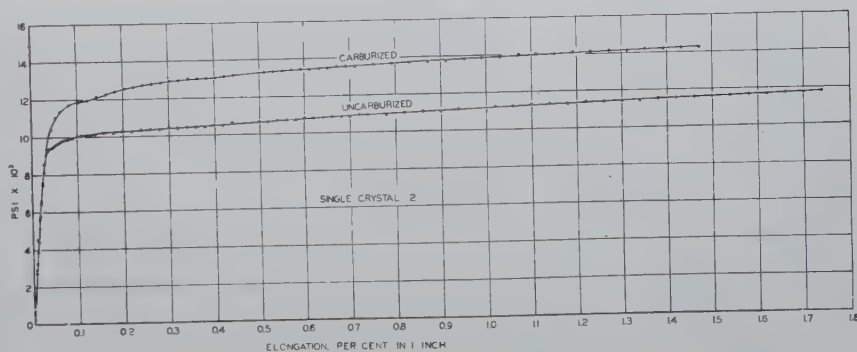


FIG 9—Stress-strain curves for single crystal 2.

aging and yield point.* The polycrystalline specimens, with one exception, were strained 10 pct before aging. In all cases, the amount of strain applied before aging was sufficient to pass through the yield point; that is, the load-elongation curve was rising steadily at the time the load was released. The amount by which the curve is raised by the aging treatment is a measure of the extent of strain aging. This was expressed as a percentage determined as follows:

$$\text{Pct strain aging} = \frac{A - B}{B} \times 100$$

where A is the yield strength in the test

* Davenport and Bain¹⁴ and Kenyon and Burns¹⁵ have shown that a marked increase in hardness occurs at this temperature in not too long an interval of time; further, that the curve of hardness vs. time has a flat maximum so that some variation in time of aging will not appreciably influence the results.

after aging and B is the stress at the time the load was released in the initial test. An exception was made in the case of the polycrystalline specimens as wet hydrogen treated. The yield strength in the test after aging was actually lower than the stress at which the load was released before aging. This was due to recovery during aging. These specimens are indicated in the results as having shown "zero" per cent strain aging. The *yield strength* was defined as the stress at which extensive plastic flow began; where a drop in load or constant load elongation was exhibited the stress at the lower yield point or at the level of constant load elongation was taken as the yield strength. As an arbitrary measure of the height of the flow curve after some strain, the stress at 4 pct strain was used. The ef-

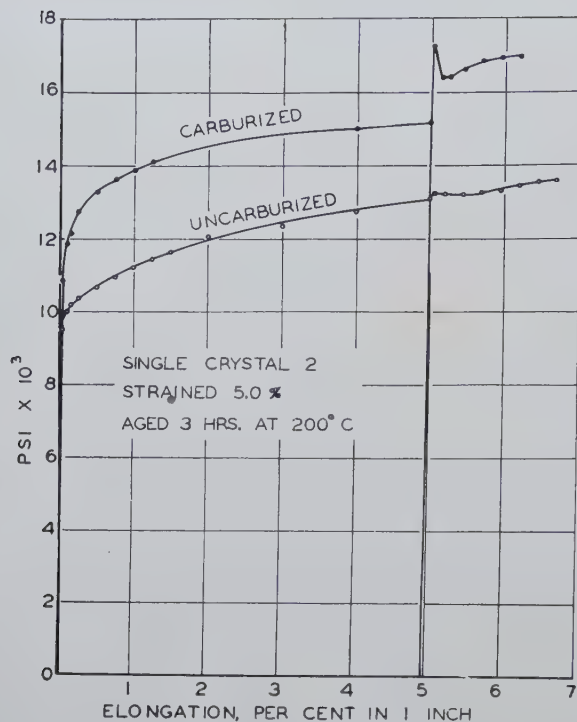


FIG 10—Composite stress-strain curves for single crystal 2.

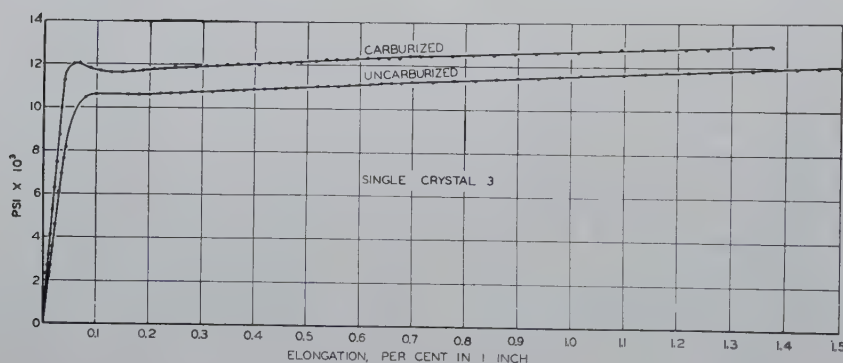


FIG 11—Stress-strain curves for single crystal 3.

fect of carburization or nitriding on the load-elongation curve is expressed as the percentage by which the treatment raised the stress at 4 pct strain. In the foregoing definition of terms the stress was computed to the nearest 500 psi based on the initial cross-sectional area. Microscopic examination of carburized and nitrided single crystals showed no evidence of a second phase.

Experimental Results

Stress-strain curves for the polycrystalline specimens are reproduced in Fig 3 to 8 and for the single crystals in Fig 9 to 20.

Some of the data derived from the stress-strain curves as well as carbon

and nitrogen analyses are tabulated in Table 2.

Discussion of Data

Examination of the stress-strain curves for the polycrystalline specimens (Fig 3 to 8) reveals results consistent with those found by Low and Genesmer.² Wet hydrogen purified material exhibited no yield point either in the initial stress-strain curve or in the test following aging. Further, no strain aging was observed. Carburizing at 700°C for 10 min. in hydrogen bearing 5 pct of normal heptane was sufficient to restore the upper yield point and some 3 pct constant load elongation at the lower yield point. The specimens

exhibited 17 pct strain aging and pronounced upper yield points in the test after aging. Nitriding at 500°C for 3 hr in hydrogen bearing 6 pct of ammonia had similar effects. These specimens exhibited upper yield points and approximately 2 pct constant load elongation. About 17 pct strain aging is observed and in the test following aging there is a constant load elongation of 2 pct; one specimen also exhibited an upper yield point in this test.

Specimens from three single crystals, 2, 3 and 4, were carburized. The uncarburized specimens from Crystals 2, 3 and 4 in every instance exhibited the following behavior: no upper yield point in the initial test nor in the test after aging; practically no constant load elongation in the initial test but 0.5 pct constant load elongation in the test after aging; no strain aging. The 0.5 pct of constant load elongation in the test after aging is consistent with the information reported by Edwards, Jones and Walters¹⁶ and Edwards, Phillips and Liu¹⁷ that low carbon steels given prolonged wet hydrogen treatment followed by slow cooling gave no yield point indication; the same samples after straining and aging gave a pronounced yield point. It was this information plus similar data for nickel, silver and copper age hardening alloys that led them to conclude that the yield point is caused by precipitation of a solute on slip planes.

Examination of the stress-strain curves of the carburized specimens of Crystals 2, 3 and 4 reveals the following information: strain aging is exhibited in every instance; Crystals 3 and 4 show a drop in load in the initial curve and Crystal 2 shows a drop in load in the test after aging; in the initial part of the curve obtained in the test after aging Crystals 3 and 4 show a low slope which rises as the test proceeds.

In summary, the uncarburized specimens of Crystals 2, 3 and 4 exhibit neither strain aging nor yield point phenomena except in the tests after aging where a slight constant load elongation was observed; the carburized specimens all show yield point indications in the initial loading as well as strain aging. Every specimen showed greater yield point indications in the test after aging than before aging.

Specimens from single Crystals 5, 6 and 7 were nitrided and the yield point and strain aging characteristics compared to non-nitrided specimens from the same crystals. It should be noted in Table 1 that the specimens cut from

Crystal 6 received insufficient wet hydrogen purification; this is evident from the yield strength and the stress level of the flow curve of the non-nitrided specimen. The non-nitrided specimens of Crystals 5 and 7 exhibited behavior similar to the non-carburized specimens of Crystals 2, 3 and 4 except for a drop in load in the initial test of Crystal 5. Examination of the stress-strain curves for the nitrided specimens reveals the following information: strain aging is exhibited in all instances, a greater amount of strain aging being observed in the nitrided specimen of Crystal 6 than in the non-nitrided specimen; a pronounced drop in load is observed in the test of Crystal 5 after aging; some constant load elongation is exhibited in most of the tests, the test of Crystal 6 after aging showing a gradually decreasing load for some 4 pct elongation after which the load rises with strain.

In summary, with the exceptions of the specimens cut from Crystal 6 which received insufficient wet hydrogen purification and a slight drop in load in the initial test of Crystal 5, the non-nitrided specimens showed no strain aging and no yield point effects while the nitrided specimens exhibit both phenomena. In the case of the exceptions noted more strain aging and yield point effect were observed in the nitrided specimens than in the non-nitrided. With one exception, greater indication of yield point behavior was exhibited in the test after aging than in the test before aging.

Addition of carbon or nitrogen had the same action, that is, to produce yield point effects and strain aging in single crystals of iron where they did not previously exist or increase these effects where they had previously existed. Aging a strained specimen, whether in the wet hydrogen purified state or carburized or nitrided, gave a greater yield point effect than in the original test.

There appeared to be no correlation between orientation and yield point behavior. A satisfactory examination of such a correlation would require a much larger number of specimens than was used in this investigation.

Microscopic evidence showed no evidence of a second phase either in the carburized or nitrided crystals. The carburized crystals contained 0.005 pct carbon and the nitrided crystals 0.007 pct nitrogen (see Table 2). The higher carbon and nitrogen contents (0.010 pct and 0.014 pct respectively) of the

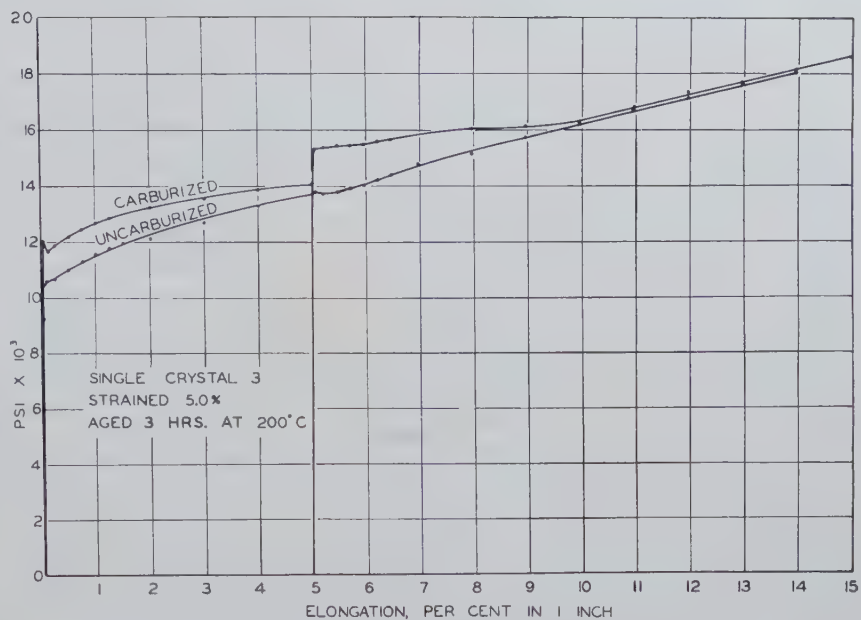


FIG 12—Composite stress-strain curves for single crystal 3.

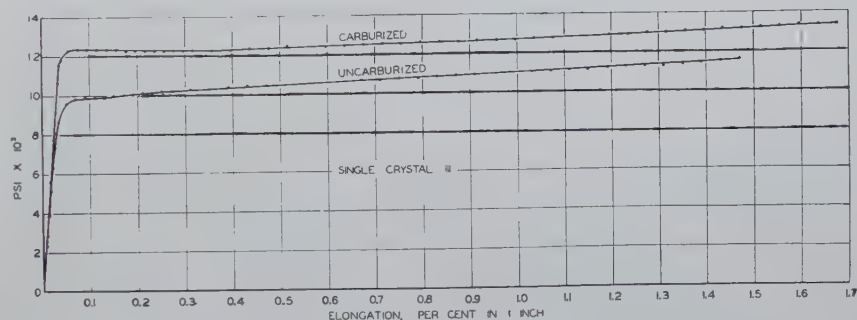


FIG 13—Stress-strain curves for single crystal 4.

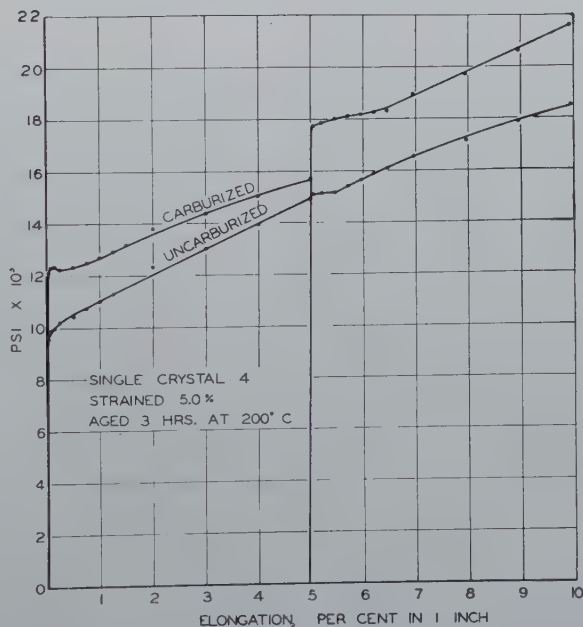


FIG 14—Composite stress-strain curves for single crystal 4.

carburized and nitrided polycrystalline specimens result from the fact that these specimens were 0.036 in. thick

while the single crystals were somewhat thicker (over 0.050 in. for the carburized crystals and about 0.043 in.

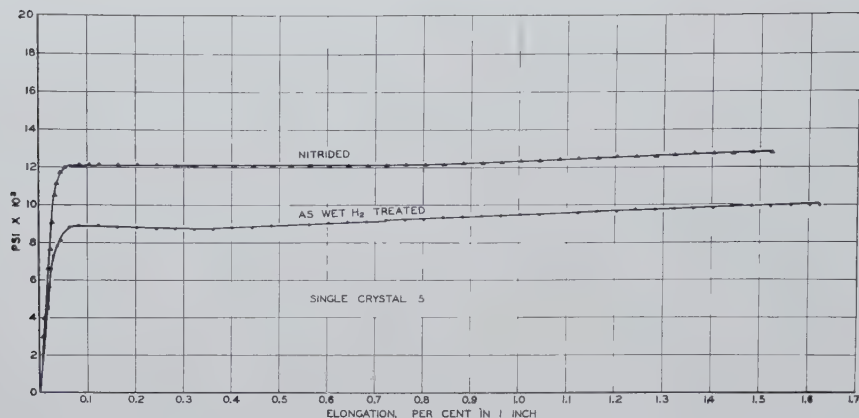


FIG 15—Stress-strain curves for single crystal 5.

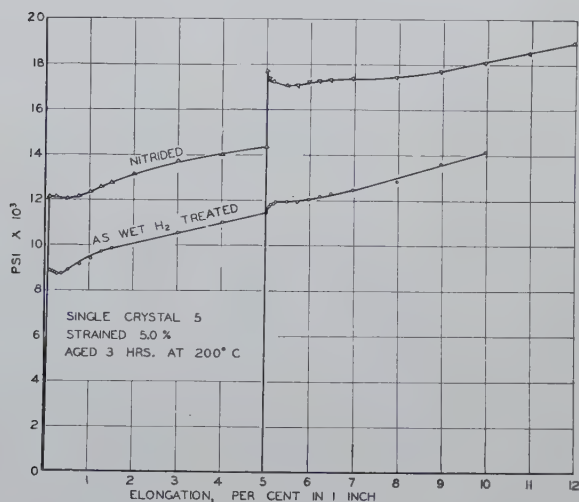


FIG 16—Composite stress-strain curves for single crystal 5.

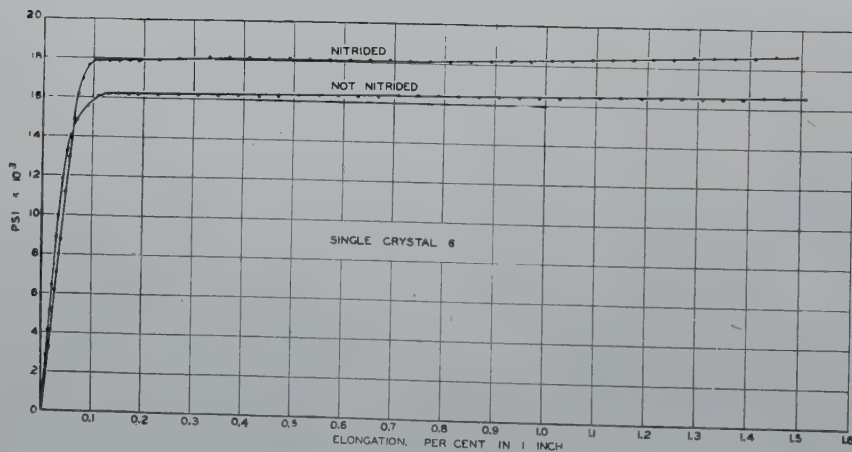


FIG 17—Stress-strain curves for single crystal 6.

for the nitrided crystals). The nitrogen analyses indicate that the polycrystalline specimens may have had higher nitrogen contents as purified than the single crystals. The presence of grain boundaries may have influenced the diffusion coefficient of carbon or nitro-

gen in alpha-iron; whether they do or to what extent is not known.¹⁸

In the light of the present data the grain boundary hypothesis as stated previously for the explanation of yield point behavior becomes untenable. Definite yield point effects have been

observed in single crystals. These effects are not as marked in the single crystals as they are in the polycrystalline specimens. Further, it has been demonstrated that single crystals which have been strained and aged show greater yield point indications after aging than before aging. This observation supports the hypothesis of Edwards, Phillips and Liu¹⁷ that the yield point is due to precipitation of solute on slip planes. Further evidence in favor of this hypothesis is the fact that mild steel as quenched exhibits no yield point but will do so if allowed to age at room temperature.^{7,19} It is suggested by the present authors that the material responsible for the yield point is in either one of two states: first, a transition state between solution and discrete second phase or, second, has just precipitated in a fine dispersion and has not had sufficient time to agglomerate. The first possibility is analogous to the transition phase which causes maximum hardness in quench aging. The second possibility was suggested by Edwards et al.

The observation is cited above that the single crystals did not exhibit yield point behavior to as great a degree as the polycrystalline specimens. It is possible that, as in quench aging, precipitation takes place preferentially at slip planes and/or grain boundaries, the latter being the more powerful in promoting yield point behavior. This would explain an increasing yield point effect with decreasing grain size as found by numerous investigators.

Conclusions

1. A yield point (drop in load and elongation at constant load) has been found in single crystals of iron containing small amounts of carbon or nitrogen. The yield point in polycrystalline iron is therefore not due to the presence of a grain boundary film as has been frequently suggested.

2. Strain aging is observed in single crystals of iron containing small amounts of carbon or nitrogen. The yield point effect is increased by strain aging in single crystals just as it is in polycrystalline irons.

Acknowledgments

The authors wish to thank Mr. F. Garofalo for his stimulating discussion and suggestions, the use of the specially

designed grips and much material assistance.

Messrs. H. F. Shannon and W. J. Frederick of the Carnegie-Illinois Steel Corporation arranged for the chemical analyses reported and their kind assistance is gratefully acknowledged.

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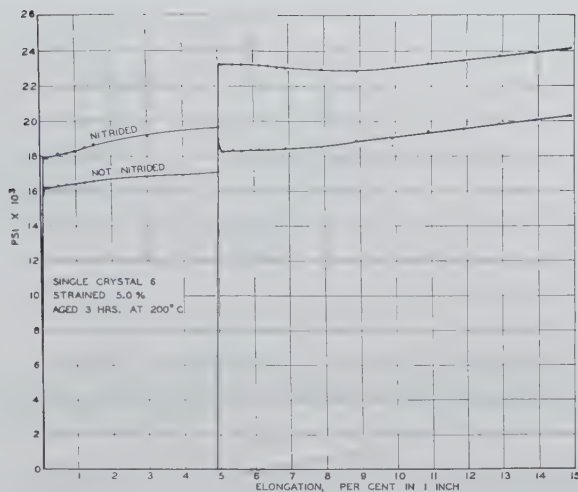


FIG 18—Composite stress-strain curves for single crystal 6.

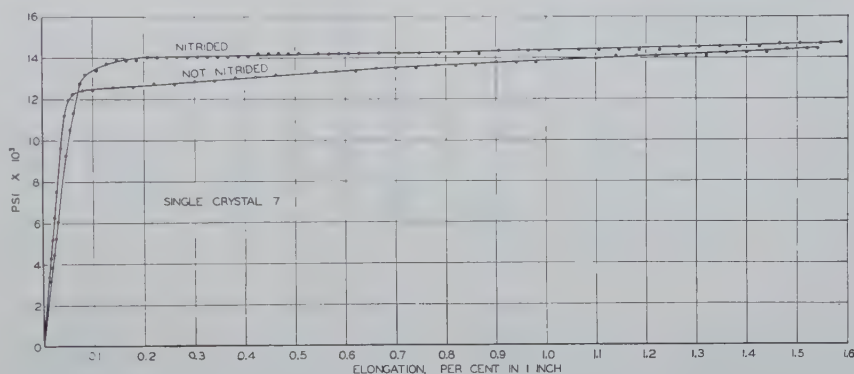


FIG 19—Stress-strain curves for single crystal 7.

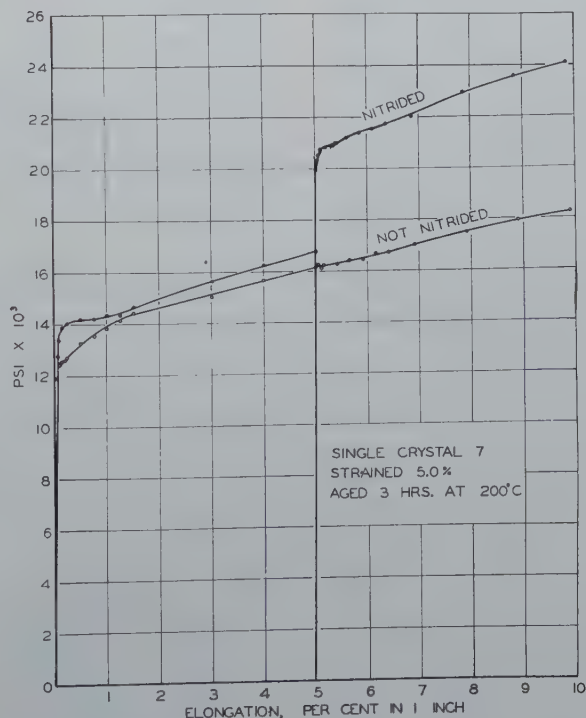


FIG 20—Composite stress-strain curves for single crystal 7.

The Effect of Oxygen, Nitrogen, and Hydrogen on Iodide Refined Titanium*

ROBERT I. JAFFEE,[†] Member AIME, and I. E. CAMPBELL[†]

Oxygen, nitrogen, and hydrogen are known to be absorbed by titanium at elevated temperatures. Ehrlich¹ reports that about 30 at. pct oxygen can be dissolved in solid solution by alpha-titanium. Nitrogen also has a solid solubility in alpha-titanium, but there is no information as to its extent. The solid solubility of hydrogen in titanium also is high. Kirshfeld and Sieverts² report 50 at. pct soluble, while Hägg³ reports about 33 at. pct hydrogen to be soluble in alpha-titanium.

General statements are made in the literature that oxygen, nitrogen, and hydrogen embrittle titanium, but no quantitative measurements have been made of the effect of these gases on the mechanical properties of the metal. One reason for this is the lack of a source of titanium pure enough to be considered a suitable starting material. Iodide-refined titanium, made by thermal decomposition of titanium iodide vapor on an incandescent wire,⁴ contains small amounts of carbon, silicon, iron, and aluminum, but is very low in oxygen, nitrogen, and hydrogen content, and, therefore, was used in this work.

A further reason for the lack of quantitative information on the effect of these gases, particularly oxygen, is the difficulty of carrying out accurate analytical determinations at the low concentrations of interest in this work.

For this reason, the oxygen, nitrogen, and hydrogen were added in measured amounts as gases, and diffused into the titanium, to avoid the necessity of chemical analysis.

The purpose of the work reported here was to isolate and measure the effects of oxygen, nitrogen, and hydrogen on titanium of the highest purity available at the time. The concentrations studied were 0.25, 0.5, and 1 at. pct in each case.

Experimental Work

One-half of a 24-in. hairpin of iodide-refined titanium was available for this work. The rod was approximately

0.25-in. in diam and had a 0.003-in. tungsten core. From chemical and spectrographic analyses, the purity of the sample was estimated to be between 99.80 and 99.95 wt pct. The chief impurities (aside from the tungsten core) were iron (0.04 pct), silicon (0.01–0.1 pct), aluminum (0.05 pct), carbon (0.03 pct). The tungsten core constituted 0.05 pct by weight of the sample. Although some alloying undoubtedly took place between the tungsten and the titanium during the deposition reaction, the core remained substantially intact in the deposited metal.

The initial average hardness of the titanium as deposited was 105 Vickers (10-kg load). The maximum hardness was 116 Vickers and the minimum 91 Vickers. The metal was somewhat harder than that currently being obtained by the iodide process, that is, 75–90 Vickers.

The rod was cold swaged to 1/4-in. diam and then cold rolled in diamond shaped rolls to about 0.080-in. square, using reductions of 15 pct in area per pass. The 0.080-in.-square wire was cut into 6-in. lengths for the preparation of the alloys by the gaseous absorption and diffusion method.

ADDITION OF OXYGEN, NITROGEN, AND HYDROGEN TO TITANIUM

A modified Sieverts' absorption apparatus, shown in Fig 1, was used in

Cleveland Meeting, October 1949.

TP 2681 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received May 5, 1949.

* The work covered in this paper was done at Battelle Memorial Institute for The Rand Corporation and the U. S. Air Force in an appraisal of titanium as a material for aircraft applications. Part of the data contained in this paper was summarized in a general article on the Battelle titanium alloy work by H. C. Cross, given at the Office of Naval Research Symposium on Titanium on Dec. 16, 1948, and also given in *Metal Progress*, (1949) 55, 356–358.

[†] Battelle Memorial Institute.

¹ References are at the end of the paper.

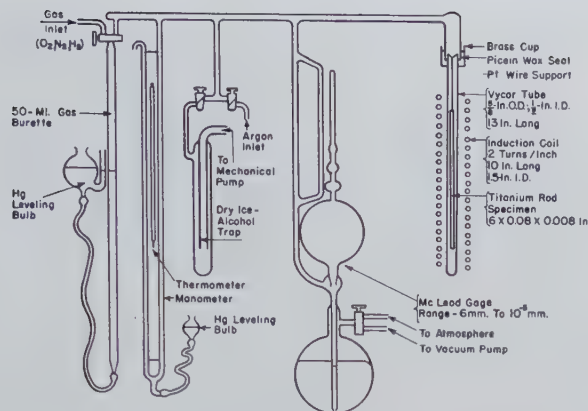


FIG 1—Gas-absorption and diffusion apparatus for producing alloys of iodide titanium with oxygen, nitrogen, and hydrogen.

adding each of the gases to the titanium samples.

Measured quantities of gas at known temperature and pressure were added to the evacuated system in each case, and the desired alloys were prepared by absorption and diffusion of the gas in the heated metal.

The samples were pickled for about 5 min. in hot, 10 pct sulphuric acid, then washed, dried, and rinsed successively in carbon tetrachloride, absolute alcohol, and acetone.

The samples were weighed to ± 0.0001 g and then suspended in an induction furnace. A No. 34 B and S ga. platinum wire was used to hold the sample.

The gas burette was flushed thoroughly with the gas to be used and then filled. Pure oxygen was obtained by slowly heating a 4:1 (by weight) mixture of C.P. potassium chlorate and manganese dioxide. The oxygen was passed through ascarite to remove traces of carbon dioxide, and then

through a dry-ice alcohol trap and magnesium perchlorate to remove water vapor. Tank nitrogen (99 pct) and tank hydrogen (99+ pct) were used as sources of those gases. Both were purified by being passed over copper turnings at 700°C and then dried in the same manner as the oxygen.

After the sample was sealed in place, the apparatus was evacuated, filled with argon, and re-evacuated. This process was repeated, but the sample tube was flamed during the final evacuation to remove occluded gas. The required volume of gas (calculated from the sample weight, barometric pressure, and temperature) was then admitted to the system from the burette and the pressure within the system redetermined. The induction-heating coil was energized, and the sample maintained at the desired temperature until the gas was completely absorbed. Additional "soaking" periods were used to obtain uniform distribution of the gas through the sample.

Data on the thermal treatment of the samples are summarized in Table 1. The oxygen was absorbed at 1000°C (observed temperature). Absorption was quite rapid and was virtually complete in 5–10 min. The samples were maintained at 1000°C for 60–80 min., however, to obtain uniform distribution of the oxygen throughout the sample.

The nitrogen was also absorbed at 1000°C but the absorption rate was much slower, 1.5–2 hr being required for complete absorption at the higher concentrations. These samples were removed as soon as absorption was complete, and given separate diffusion treatments at 1075–1100°C in vacuo.

Hydrogen, unlike nitrogen and oxygen, is absorbed reversibly, and at the higher concentrations, absorption takes place more rapidly at lower temperatures. At 650–700°C, concentrations up to one atomic per cent were completely absorbed in 2–4 min. Additional "soaking" periods were used as indi-

Table 1 . . . Identity of Specimens

Specimen No.	Addition	Composition		Thermal Treatment	Density, g per cc	Electrical Resistivity at 25.6°C Microhm-cm
		At. Pct	Wt Pct			
1	Control	Zero	Zero	60 min. 950–1070°C in vacuum	4.53	48.5†
12		Zero	Zero	60 min. 995–1010°C in vacuum	4.52	47.7
13		Zero	Zero	60 min. 1090–1125°C in vacuum	4.515	47.8
2	Oxygen	0.27	0.0905	60 min. 1000°C in vacuum	4.52	50.4
3		0.51	0.171	60 min. 1000°C in vacuum	4.51	51.8
4		1.01	0.34	60 min. 1000°C in vacuum	4.515	55.6
7	Nitrogen	0.245	0.0717	50 min. 1100–1110°C in vacuum*	4.53	49.75
6		0.496	0.146	47 min. 1075°C in vacuum*	4.53	52.95
5		0.996	0.293	90 min. 1080°C in vacuum*	4.525	55.8
10	Hydrogen	0.27	0.00569	60 min. 1000°C	4.56	47.2
9		0.50	0.0106	15 min. 1000°C, 15 min. 775–800°C, 30 min. 650–700°C	4.525	48.1
8		0.99	0.021	30 min. 1000°C, 25 min. 900°C, 15 min. 750°C, 60 min. 600–700°C	4.515	47.8

* This comprises only the diffusion treatment for the Ti-N specimens, and does not include the absorption treatment.

† Specimen No. 1 was harder than either Specimen 12 or 13, and, therefore, may not be considered as pure as those specimens. The values of 47.7 and 47.8 microhm-cm probably are better values for iodide-refined titanium.

cated in Table 1 to insure complete diffusion.

DISCUSSION OF ABSORPTION

When large amounts of oxygen or nitrogen are admitted to the absorption chamber, oxide or nitride films are formed, which act as barriers that retard the absorption process. Slow continuous addition of these gases at low pressures, therefore, gives higher overall absorption rates than step-wise additions at higher pressures. In the case of nitrogen, which is absorbed slowly, the last traces of gas may be only adsorbed, rather than absorbed, but if the sample is maintained at temperature for a few minutes, the adsorbed gas will diffuse into the metal.

Hydrogen, unlike nitrogen and oxygen, is best absorbed at higher pressures, and, for higher concentrations than those reported here, the procedure should be modified to the extent that a measured fraction of this gas should be absorbed, rather than the entire addition. At higher hydrogen concentrations, an appreciable equilibrium pressure of hydrogen is developed at temperatures where the absorption is quite rapid, and to take advantage of the rapid absorption at the higher temperatures, the hydrogen should be added at pressures up to one atmosphere and the extent of the hydrogen absorption regulated by controlling the absorption time.

The absorption technique used in this work provides an accurate method for adding precise quantities of hydrogen, nitrogen, and oxygen to titanium, and where the limits of solid solubility are not exceeded, homogeneous alloys can be obtained in reasonable diffusion times. Weight changes obtained upon addition of these gases to the titanium check well with the calculated weights of the gases added.

Since the amounts of gases added in this work were small, the evaporation of titanium did not introduce a significant error. The loss of titanium was insignificant in itself, and for short

absorption times the absorption of gases by titanium vapor and volatilized titanium can be neglected.

The specimens prepared were 6 in. long and approximately 0.080-in. square. Their compositions and physical condition are summarized in Table 1. For convenience, in the rest of this report the nominal compositions, 0.25, 0.5, and 1 at. pct, will be referred to, rather than the actual composition.

From each 6-in. specimen, a length 4.25 in. long was cut for determination of density, cross-section, and electrical conductivity. This was then cut into two pieces, one 3 in. long for a tensile test, and the other about 1 in. long for a cold-rolling test. The remainder of the original specimen was reserved for microscopic examination.

DENSITY, RESISTIVITY

Density at room temperature was measured by determining the displacement of the specimen, 4.25 in. long, in distilled water at a known temperature.

Results of density measurements are tabulated in Table 1. The cross-sections of the specimens were determined by calculation from the weight, length, and density of the specimens. Resistance of a 3.5-in. length of the specimen was determined at 25.6°C (78°F) by means of a Kelvin bridge. Values of the electrical resistivity were calculated from the resistance, cross-section, and length, and are given in Table 1

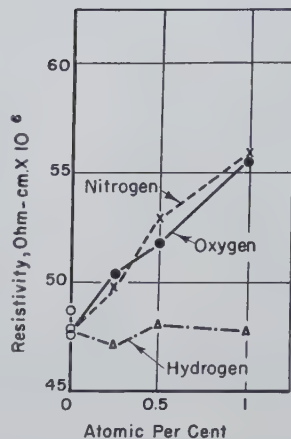


FIG 2—Effect of oxygen, nitrogen, and hydrogen on the electrical resistivity of iodide titanium.

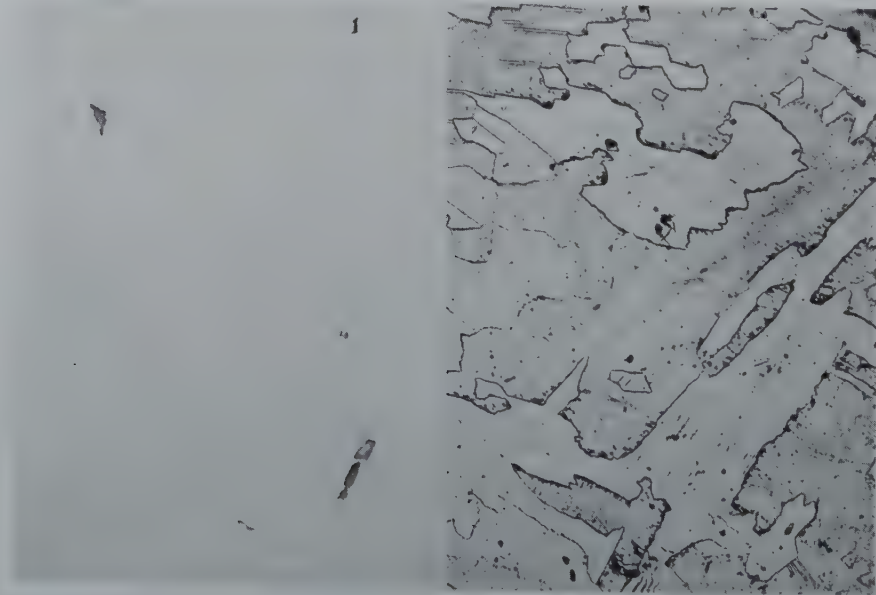


FIG 3—Unalloyed titanium. Unetched. 1000 X.

FIG 4—Unalloyed titanium. Annealed at 1000°C. 250 X.

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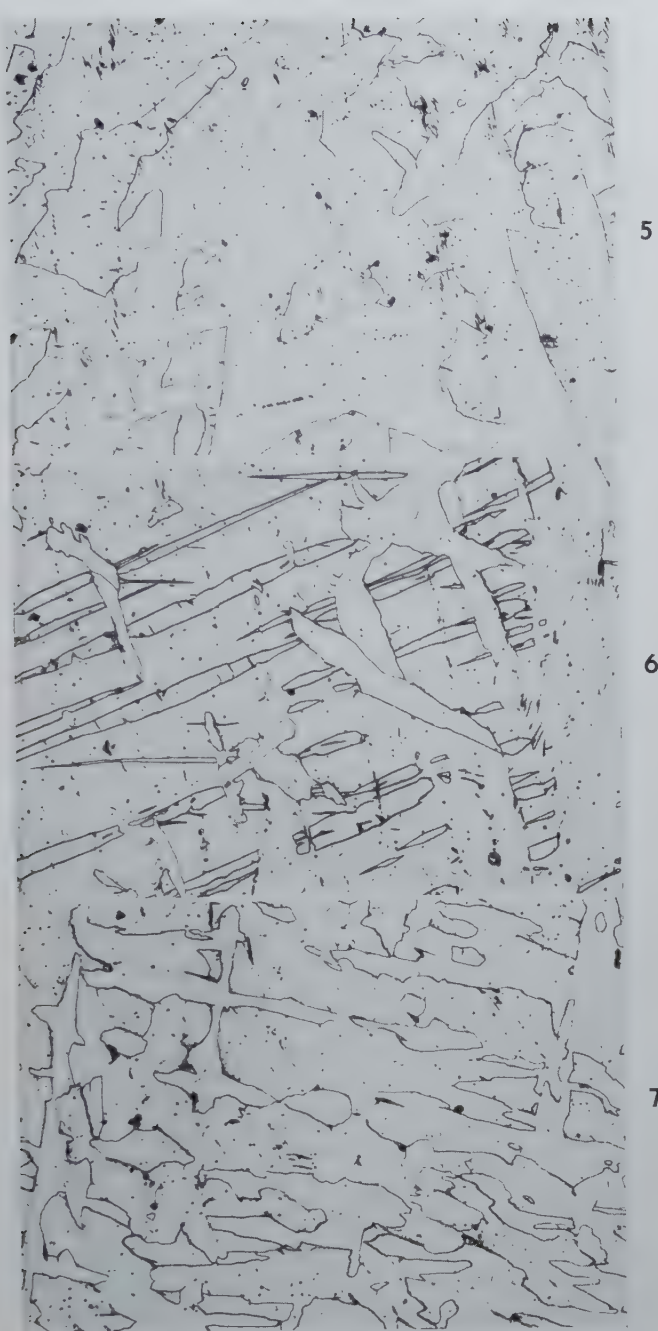


FIG 5—0.25 at. pct oxygen. Annealed at 1000°C. 250 $\times$.
 FIG 6—0.5 at. pct oxygen. Annealed at 1000°C. 250 $\times$.
 FIG 7—1 at. pct oxygen. Annealed at 1000°C. 250 $\times$.

Figures reduced one-seventh in reproduction.

There is no significant change in the density of the alloys containing oxygen and nitrogen with alloy content. There may be a change in density with hydrogen content, as indicated by the high value for the 0.25 at. pct H alloy in Table 1, but this is rather doubtful. Most of the densities fall between 4.51 and 4.53, which are in excellent agreement with the values reported in the literature.

A plot of the electrical resistivity data is shown in Fig 2. Both oxygen and nitrogen raise the resistivity of titanium to the same extent per atomic per cent, the increase in resistivity being almost linear. This is in agreement with the reported solid solubilities of these elements in alpha-titanium. Hydrogen to one atomic per cent does not appear to affect the electrical resistivity.

The values of 47.8×10^{-6} ohm-cm and 47.7×10^{-6} ohm-cm found for the electrical resistivity at 25.6°C are in fair agreement with the value of 47.5×10^{-6} ohm-cm at 20°C reported by van Arkel.⁵ Using the value of the temperature coefficient of resistance given by van Arkel, 0.00425 per °C, the two experimental values become 46.7×10^{-6} ohm-cm and 46.6×10^{-6} ohm-cm at 20°C. Magnesium-reduced

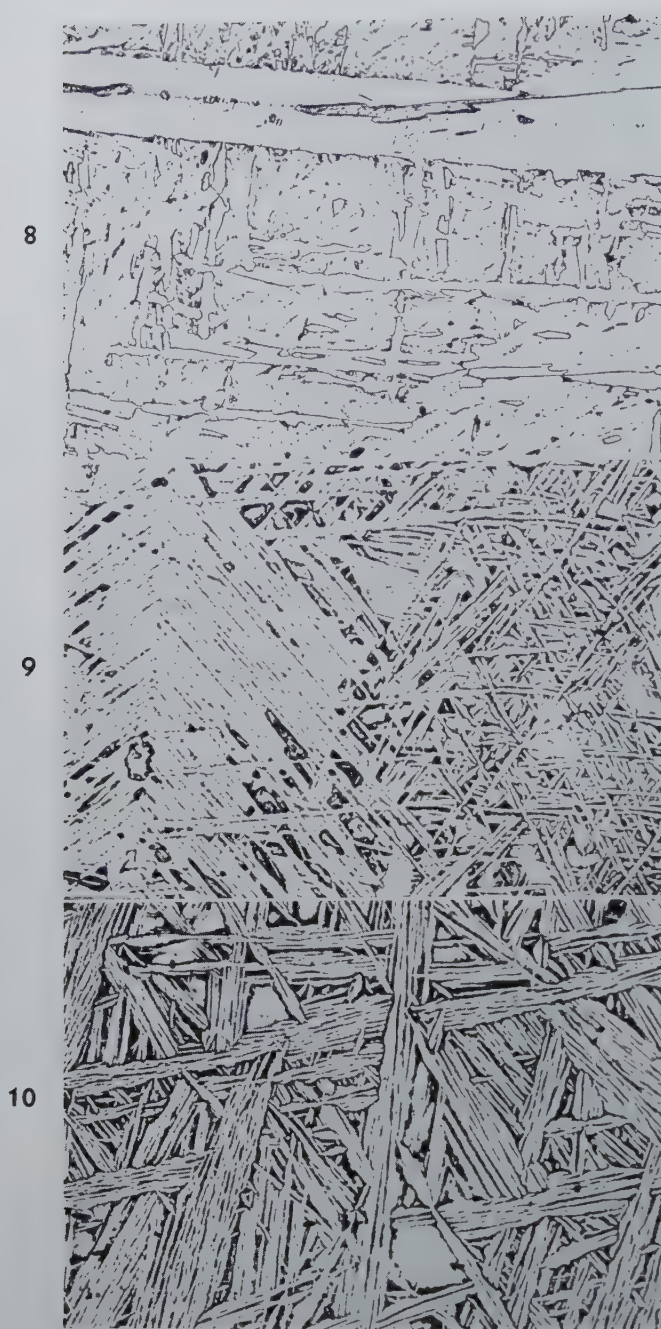


FIG 8—0.25 at. pct nitrogen. Annealed at 1100°C. 250 ×.

FIG 9—0.5 at. pct nitrogen. Annealed at 1100°C. 250 ×.

FIG 10—1 at. pct nitrogen. Annealed at 1100°C. 250 ×.
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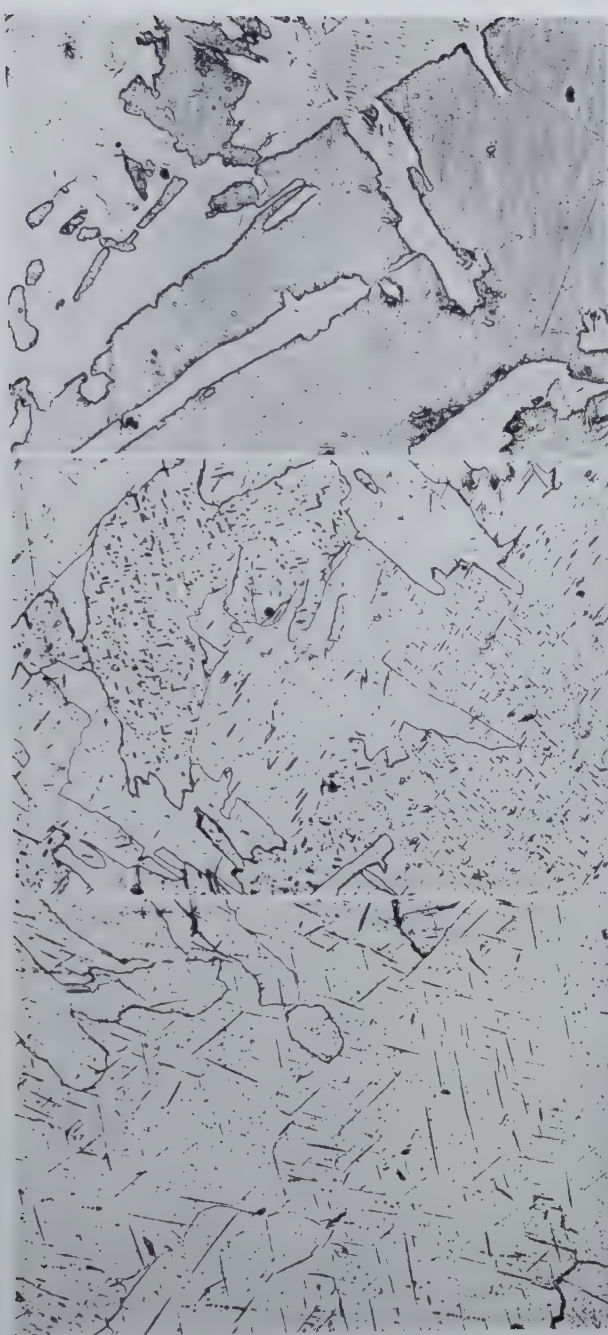
titanium has an electrical resistivity at 20°C of 56×10^{-6} ohm-cm, according to Dean, Long, Wartman, and Anderson,⁶ and 55.4×10^{-6} ohm-cm according to Greiner and Ellis.⁷ This higher resistivity is undoubtedly caused in part by dissolved oxygen and nitrogen, as indicated here by the potent effect of these elements on the electrical resistivity.

MICROSTRUCTURE

Cross-sections and longitudinal sections of each of the specimens were examined metallographically. No apparent lack of homogeneity was observed in the cross-sectional structure of any of the alloys, apart from the diffusion area around the tungsten core.

Polishing was done on wool laps

using 0-2-micron diamond abrasive suspended in carbon tetrachloride. In order to bring out a true structure on the titanium specimen and the softer of the alloys, it was necessary to polish and etch alternately three or four times to eliminate the worked surface. Etching was done by swabbing for 5-10 sec with an etchant containing 3 pct concentrated HNO_3 , 1.5 pct con-



11

12

13

FIG 11—0.25 at. pct hydrogen. Annealed at 1000°C. 250 ×.

FIG 12—0.5 at. pct hydrogen. Annealed at 700°C. 250 ×.

FIG 13—1 at. pct hydrogen. Annealed at 700°C. 250 ×.

Figures reduced one-seventh in reproduction.

concentrated HF, balance water (volume percentages).

In the as-polished condition, all of the specimens showed a number of gray inclusions. Fig 3 shows a typical field of these inclusions at 1000 × magnification. The identity of these inclusions is not known. It is suspected that they may be titanium silicide.

Unalloyed titanium in the condition as cooled from 1000°C has the

structure shown at 250 × in Fig 4. It consists of large plates of alpha-titanium arranged in a somewhat Widmanstättenic manner; the alpha plates probably formed along the octahedral planes of the high-temperature cubic phase on cooling through the transformation temperature.

The structures of alloys with 0.25, 0.5, and 1 at. pct oxygen are shown in Fig 5, 6, and 7, respectively. The

general effect of oxygen seems to be to refine the structure, making the plates of the alpha-titanium smaller, and the Widmanstätten arrangement more regular.

The structures of the nitrogen-bearing alloys are shown in Fig 8, 9, and 10. The effect of nitrogen on the microstructure is to promote the formation of needles of alpha titanium. This is a most interesting observation, be-

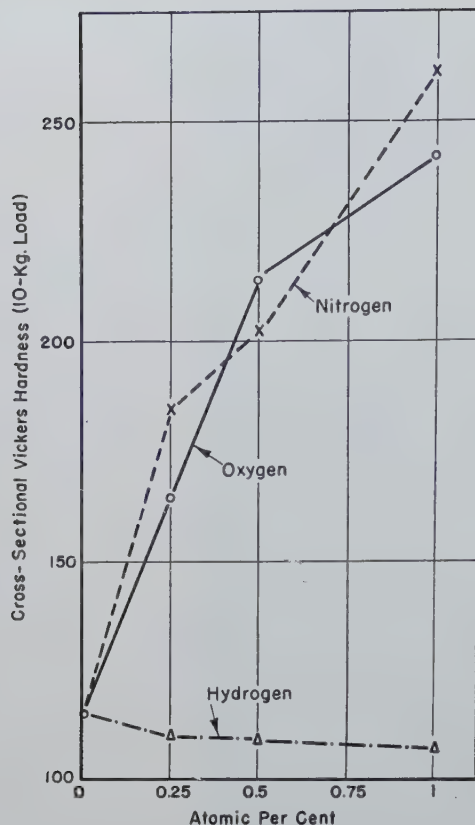


FIG 14—Average cross-sectional hardness values for alloys of iodide titanium with oxygen, nitrogen, and hydrogen.

cause this needle-like Widmanstätten structure is characteristic of most magnesium-reduced titanium that has been annealed above the alpha-beta transformation temperature. The conclusion may be drawn that the needle-like structure found in most less pure kinds of titanium is caused by nitrogen in solid solution.

The structures of the hydrogen alloys are shown in Fig 11, 12, and 13. The alloys containing 0.25 and 0.5 at. pct of hydrogen are not materially different from unalloyed titanium. They show large Widmanstätten plates of alpha-titanium. Origin of the markings shown inside the grains in Fig 12 is not known. They may be transitional structures developed by the grinding and polishing-etching operations. Fig 13, for the one atomic per cent hydrogen alloy, shows an arrangement of line markings which resembles a Widmanstätten precipitate. The origin of these marks is not known. It is doubtful that they are a second phase based on hydrogen, because one atomic per cent is very far from the accepted solid solubility of hydrogen. Another peculiar thing about them is that they cross over grain boundaries in some cases. A

possibility is that they are a kind of deformation twin, originating in the preparation of the specimen for metallographic examination.

MECHANICAL PROPERTIES

Hardnesses were taken on a cross-section of each of the specimens to see whether there was any difference in hardness between the surface and center. The only specimen for which this condition existed was No. 6, 0.5 at. pct nitrogen. Here the surface had a hardness of 245 Vickers, and the center had a hardness of 170 Vickers. This may be associated with its shorter diffusion time of 47 min. at 1075°C. Specimen No. 5, one atomic per cent nitrogen, was diffused 90 min., at 1080°C, and was uniform from center to surface.

A plot of the average cross-sectional hardness of the specimens versus their compositions is shown in Fig 14.

Tensile tests were run on 3-in. lengths of each specimen, observations being made of ultimate tensile strength and per cent elongation in one inch. Results of these tests are shown in Fig 15.

The hardness data for oxygen and nitrogen alloys are somewhat erratic, but indicate that oxygen and nitrogen

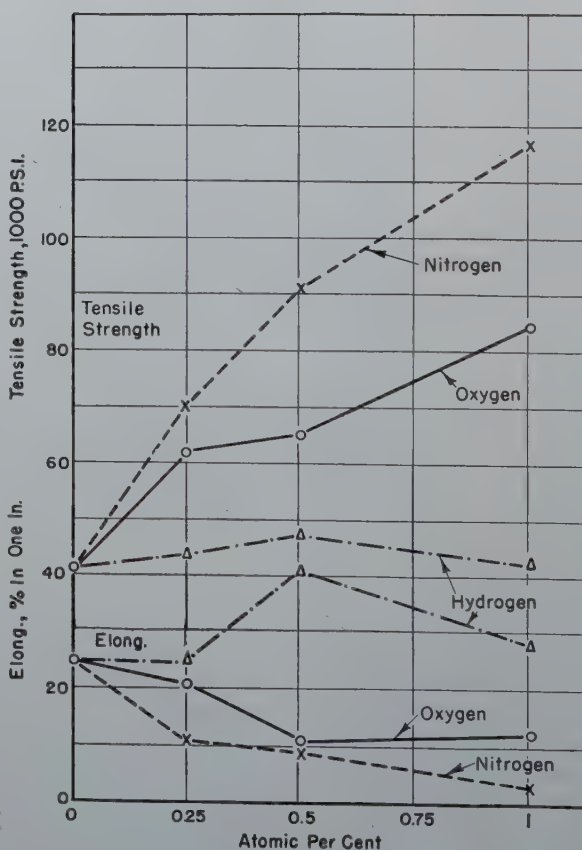


FIG 15—Tensile strength and elongation of alloys of iodide, titanium with oxygen, nitrogen, and hydrogen.

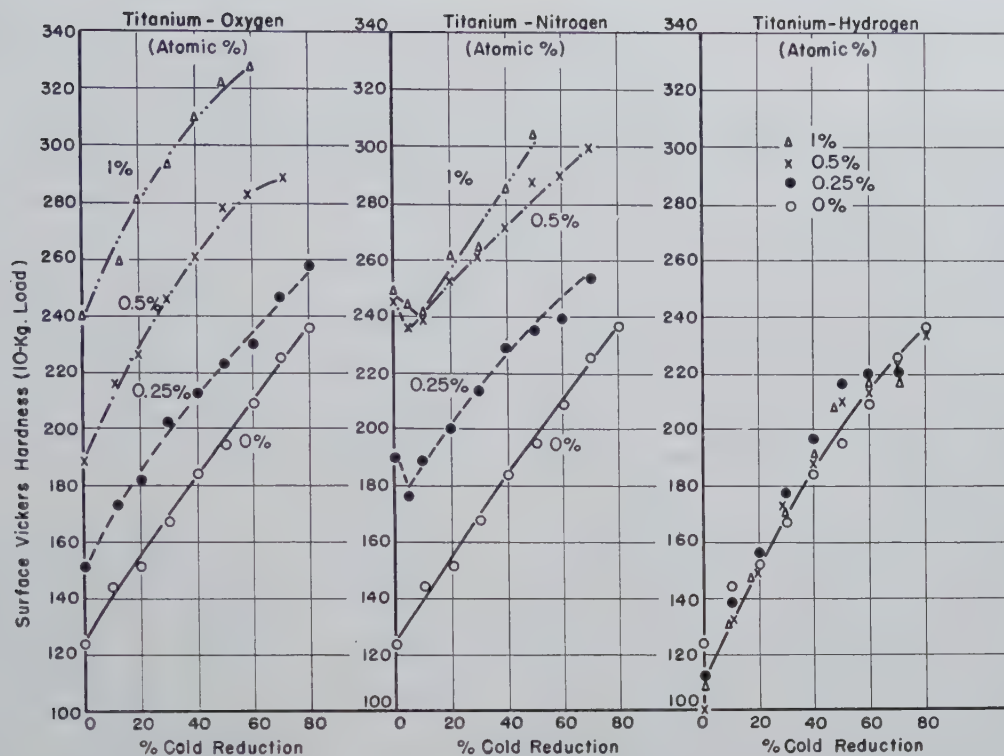


FIG 16—Work-hardening curves of iodide titanium and titanium-oxygen, titanium-nitrogen, and titanium-hydrogen alloys.

Table 2 . . . Cold Reductions Causing Edge Cracking

Specimen No.	Composition, Atomic Per Cent	Initial Hardness, Vickers (10-kg load)	Rolled to, Inch	Per Cent Reduction	Edge Cracked
12	100 Ti	116	0.004	95	No
2	0.25 O	164	0.004	95	No
3	0.5 O	214	0.024	70	Yes
4	1 O	242	0.032	60	Yes
7	0.25 N	184	0.040	50	Yes
6	0.5 N	202	0.032	60	Yes
5	1 N	262	0.046-0.048	30-40	Yes
10	0.25 H	110	0.004	95	No
9	0.5 H	109	0.004	95	No
8	1 H	107	0.004	95	No

harden titanium to about the same extent, with nitrogen possibly having a greater effect. The positions of oxygen and nitrogen are reversed at the 0.5 at. pct composition, and it is believed that this is not the actual case. As was pointed out in previous discussion, the 0.5 at. pct nitrogen alloy was incompletely diffused, and the soft center probably was given too much weight in the straight arithmetic average taken of the cross-sectional hardnesses.

Hydrogen up to one atomic per cent does not increase the hardness of iodide titanium, and actually appears to decrease it slightly, although the apparent decrease may have been caused by hardness variations in the

iodide titanium rod itself.

Nitrogen has a considerably greater strengthening effect on titanium than oxygen when added in concentrations of up to one atomic per cent, and correspondingly decreases the ductility to a greater extent. The hydrogen alloys appear to be slightly stronger and more ductile than the titanium control specimens. Since the elongation of pure titanium is generally found to be closer to 40 pct in one inch, rather than the 30 pct average found with the control specimens, the high ductilities of the hydrogen-containing specimens are more likely the result of specimen variation rather than any interaction of hydrogen with the impurities in the

titanium. Thermodynamic considerations indicate that reactions between hydrogen and the residual oxygen, nitrogen, or carbon in the titanium would not proceed to an appreciable extent under the conditions of preparation of the hydrogen-containing alloys.

COLD ROLLING CHARACTERISTICS

One-inch lengths of each of the 0.080-in.-square specimens were cold rolled at 0.001 in. per pass until edge cracking started, or to the minimum thickness the mill could roll, which was 0.004 in. Surface Vickers hardnesses (10-kg load) were taken at steps of 10

pct reduction in thickness to 70 or 80 pct reduction if the cold rolling could be carried that far.

The reductions at which edge cracking became apparent are shown in Table 2 for the various specimens.

Pure titanium and the Ti-H alloys all were very soft initially and cold rolled to 0.004 in. or 94 pct reduction without edge cracking. The 0.25 at. pct oxygen alloy, which was 164 Vickers initially, also could be rolled to 95 pct reduction without edge cracking. A completely different result was found with the 0.25 at. pct nitrogen alloy, which was 184 Vickers originally, only slightly higher than the oxygen alloy. It could be rolled only to 50 pct reduction before edge cracking. This indicates that nitrogen content is more deleterious to cold rollability than oxygen content of either the same amount or the same hardness. This adverse effect of nitrogen compared to oxygen on cold rollability shows up also in the edge-cracking limits at the 0.5- and 1-at. pct levels.

WORK-HARDENING CURVES

The work-hardening curves found for the various alloys are plotted in Fig 16.

The work-hardening curve of the unalloyed titanium (Specimen No. 1) is approximately a straight line. An 80 pct reduction about doubles the original hardness. The strip was rolled to 95 pct reduction, but determinations of hardness values were stopped at 80 pct.

Oxygen alloys had work-hardening curves very similar to the unalloyed titanium, apart from the different hardness levels from which the alloys start. This means that the relative ratios of hardness to the Ti-O alloys to titanium are unaffected by cold working.

The work-hardening data of the Ti-N alloys had some peculiarities. First, there was an initial decrease in the hardness, which was followed by

a normal increase in hardness on further cold working. This anomalous decrease in hardness may have been caused by a hard surface shell of nitrogen-rich material, which, once thinned out by a cold reduction, permitted the hardness indenter to penetrate to the softer core. The second peculiarity of the work-hardening curves of the Ti-N alloys is the closeness of the curves for the 0.5 at. pct and 1 at. pct nitrogen alloys. This does not seem to be in line with the results of the tensile tests. Actually, since the 1 at. pct nitrogen alloy only had 3 pct elongation and showed signs of edge cracking at 30 pct reduction, there probably were not enough data obtained on sound metal from which to draw conclusions. The normal part of the curve for the 0.25 at. pct nitrogen alloy shows about the same characteristics as unalloyed titanium, with the hardness level about 15 Vickers points above the curve for the 0.25 at. pct oxygen alloy.

The work-hardening data for the titanium hydrogen alloys were so similar to unalloyed titanium that no attempt was made to separate them in Fig 16.

Summary

Oxygen and nitrogen up to one atomic per cent increase the electrical resistivity at 25.6°C linearly from 48 to 56 micro-ohm-cm, while hydrogen in the same concentration has no effect.

Dissolved nitrogen changes the microstructure of annealed titanium from large Widmanstätten plates to long, narrow Widmanstätten needles. Oxygen refines and makes the Widmanstätten arrangement of the alpha-Ti plates more regular. Hydrogen does not change the microstructure of titanium at 0.25 at. pct concentration, but at higher concentrations, some markings of unknown origin appear.

Nitrogen has a more potent hardening and strengthening effect than oxygen and decreases the ductility to a greater extent. Hydrogen, when added

to one atomic per cent either does not affect the mechanical properties, or slightly improves both the strength and ductility.

Unalloyed titanium, the titanium-hydrogen alloys, and the 0.25 at. pct oxygen alloy could be cold rolled to 95 pct reduction without edge cracking. The higher oxygen alloys edge cracked at 60-70 pct reductions, while the nitrogen-containing alloys had considerably less capability for cold rolling without edge cracking. Work-hardening curves were determined for the various specimens.

Acknowledgment

Acknowledgment for support and permission to publish this paper is hereby extended to the U. S. Air Force and The Rand Corporation. The authors are grateful to Dr. B. W. Gonser for helpful discussions in planning and evaluating the work.

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The Origin of Annealing Twins in Brass

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According to conventional crystal mechanics, a face-centered cubic single crystal slips on the system $\{111\} \langle 110 \rangle$ which receives the highest shear stress in terms of the stated orientation of the stress axis of the crystal.¹ However, in brass, the slip process is never as simple as this conventional analysis indicates. There is always a second cooperating plane which contains the same slip direction as the primary plane.^{2,3} There is still another cooperating slip system commonly called the conjugate system³ previously observed to function with considerable lag only after the rotation due to the action of the first plane had produced a symmetrical condition of equal shear stress on the two systems.

In the 1943 Campbell Lecture,⁴ a slip mechanism was outlined in which the $\{111\}$ planes moved step-wise in two adjacent $\langle 112 \rangle$ directions integrating into the $\langle 110 \rangle$ slip direction. "... the slipping process would be visualized as a succession of steps 30° to the right and left of the resultant, microscopically observed, slip direction; a first step setting up the twinned configuration in the single spacing involved and an ensuing slip returning the atoms to the untwinned configuration while completing a fully integrated $\langle 110 \rangle$ movement."⁴ This process is shown diagrammatically in Fig 1 which shows two close packed layers of atoms at a slip site. A movement of the full circles through the "valleys" into the adjacent sites would produce a twin configuration with respect to the open circles. The term, "twin fault," seems most appropriate to define this faulted condition of the lattice.

Annealing twins in face-centered cubic metals may arise from preformed nuclei which could be described as twin faults produced by the first movement of this two stage slip process.

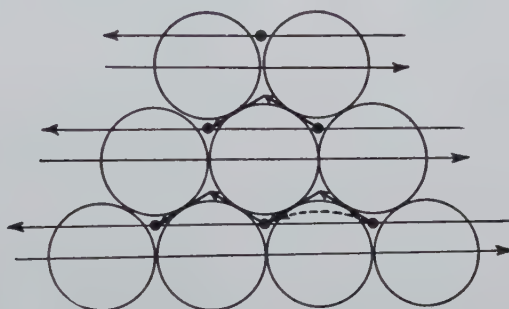


FIG 1—Hypothetical mechanical twin formation by deviation of slip from "foothills" into "valleys" of close-packed face-centered cubic structure.

(Mathewson⁴)

However, it is clear that this kind of slip does not take place so as to embrace a measurable number of adjacent planes because careful X ray examination has not conclusively shown the existence of ponderable twin lamellae (as in the case of Neumann bands in the body-centered ferrite).

In view of the imponderable character of these hypothetical twin faults, the search for evidence of their existence seems restricted to indirect methods.

The most obvious experimental technique serving this purpose is the determination by micrographic and X ray methods of the alignment of the composition planes of annealing twins with

the former slip planes in axially strained and recrystallized single crystals. In previous attempts of this sort not all of the annealing twins could be traced to slip planes known to be operative in the conventional sense and hence evidence appeared negative or inconclusive. With the discovery of additional slip systems^{2,3} operating in conjunction with the major system of highest resolved shear stress in axially strained brass it became apparent that the simple technique outlined above might be re-employed with more gratifying results.

If, on annealing single crystals of alpha brass previously strained in tension, twins are found with their composition planes parallel to all three operative slip planes and with no participation of the fourth plane there is good evidence in favor of the existence of twin faults and of the two stage slip process.

Experimental Procedure and Results

Single crystals of alpha brass of the 70-30 composition were grown by solidification from the molten state.

Cleveland Meeting, October 1949.
TP 2676 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before December 1, 1949. Manuscript received May 2, 1949.

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¹ References are at the end of the paper.

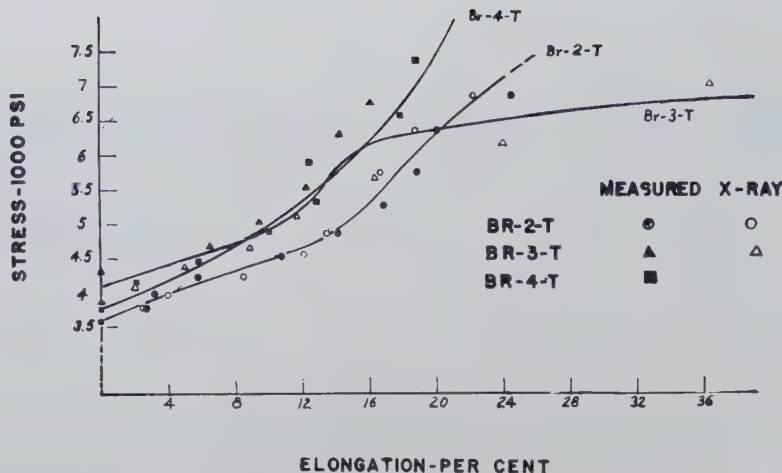


FIG 2—Stress-elongation curves for three tapered single crystals.

After a long homogenization treatment (24 hr at 800°C), central sections, about 3 in. long, were carefully tapered in a lathe to provide a linear decrease in diameter from 0.5 to 0.32 in. along the gauge length. Etching in 1:1 aqueous nitric acid solution removed the cold work, and subsequent electrolytic polishing² provided a smooth metallographic surface. Back-reflection Laue photograms were made at four different radial positions to insure that all cold work had been removed and to determine the orientation of the crystals. Fine black lines were painted on each specimen at one-quarter inch intervals; these intervals were carefully measured on a Zeiss metallograph at a magnification of X77.

The crystals were extended with a constant rate of loading until slip lines became visible within one-half inch of the large end of the gauge length. The average of repeated measurements of the gauge intervals using the same magnification gave the elongation per one-quarter inch interval. Stress values were derived from the maximum load to which the crystals were extended and the areas of the specimen after extension. A series of four back-reflection Laue photograms per gauge interval gave reorientation values in terms of λ and χ , where λ is the angle between the specimen axis and the primary slip direction and χ is the angle between the specimen axis and the primary slip plane. These values were used to calculate the elongation in the usual manner,⁵ viz.

$$\frac{l_1}{l_0} = \frac{\sin \chi_0}{\sin \chi_1} \quad [1]$$

where χ_0 and χ_1 are the original and final angles, and l_0 and l_1 are the original and final gauge lengths. The data relating stress to the measured and

calculated elongation are plotted in Fig 2. In the case of Specimen BR-4-T only measured values are shown because the reorientation was too small to yield reliable calculations.

Using the values of λ and χ determined by means of X rays, shear stress resolved in the primary $\langle 110 \rangle$ direction of slip and on the primary plane of slip was calculated by means of the usual formula.⁵

$$S = \frac{P}{A} \sin \chi \cos \lambda \quad [2]$$

where P is the load; A , the area; S , the resolved shear stress; and χ and λ , as previously designated. The shear was calculated from the two expressions;

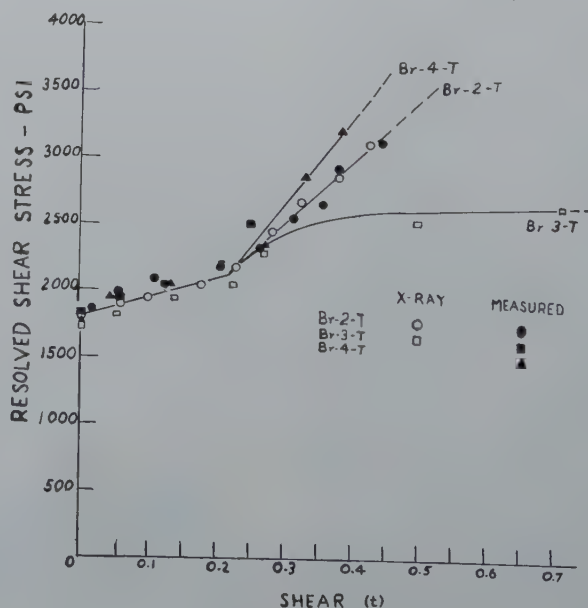


FIG 3—Resolved shear stress-shear curves for tapered single crystals.

$$t = \frac{\cos \lambda_1}{\sin \chi_1} - \frac{\cos \lambda_0}{\sin \chi_0} \quad [3]$$

and

$$t = \frac{1}{\sin \chi_0} \left(\sqrt{\left(\frac{l_1}{l_0} \right)^2 - \sin^2 \lambda_0} - \cos \lambda_0 \right) \quad [4]$$

Formulas 2 and 3 gave resolved shear stress-shear values from the X ray data and formulas 1, 2 and 4 gave resolved shear stress-shear values from the measured data. These resulting shear strengthening curves are shown in Fig 3.

The rotations of the lattice, Fig 4, are plotted from the back reflection X ray determinations of the orientations along the tapered gauge length.

These tapered specimens afforded an excellent opportunity to study the progressive formation of slip lines because of the increasing amount of deformation from zero per cent at one end to beyond 24 pct at the other. A series of representative micrographs of slip lines in Spec. Br-2-T is shown in Fig 5-16 together with the approximate extension at the respective position on the tapered specimen.

The first observable slip line, Fig 5, appeared very thin at X500. Even at this initial stage, slip on the conjugate plane is evident. Cross-slip, although not shown in Fig 5, was nevertheless present in the first group of slip lines at a different point of observation (cf Fig 17) and is shown in Fig 10 after an

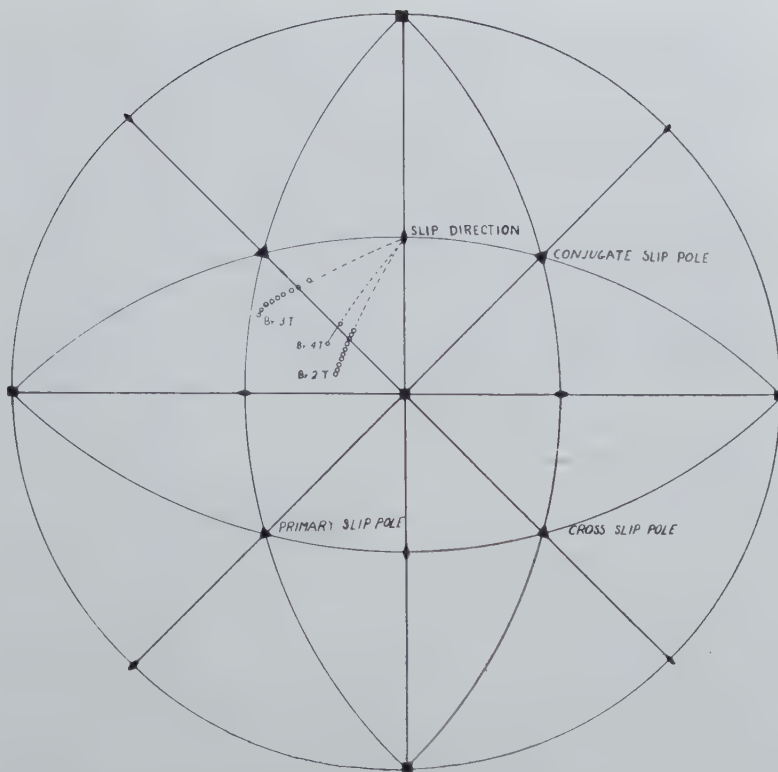


FIG 4—Stereographic projection showing the axes rotations of the tapered single crystals pulled in tension.

elongation of 4 pct. With increasing deformation, new slip lines appear adjacent to former lines, in such locations as to widen the original band, and produce the familiar clustered appearance.

At about 4 pct elongation (Fig 10) the clusters are sensibly wide and at the higher deformations, perhaps beginning with about 16 pct elongation, the clusters have overlapped to form a continuous bundle of lines which have lost their sharpness because of the proximity of many lines in and out of sharp focus.

The specimens were next wrapped loosely in thin, dead soft, brass foil of the same composition and annealed for

one hour at 800°C. After furnace cooling, they were etched in nitric acid solution followed by ferric chloride. Microscopic examination revealed that the composition planes of the many twins were nearly parallel to one of three different slip planes of the parent lattice. This structure is shown in Fig 18 and 19. The genesis of the twins was proven by X ray examination in which the X ray beam was positioned partly on the twin and partly on the adjacent material. Crystallographic analysis of the resulting double set of Laue spots gave the stereographic plot shown in Fig 20 which is typical of many such analyses made to establish the twin

relationships.

In all three specimens, recrystallization was found to extend to the location representing a minimum value of shear of about 0.22 (see Fig 3). The amount of recrystallization observed in Spec. Br-3-T was small in comparison detected in the other two specimens. Nevertheless, grains in all three specimens were large and replete with annealing twins, all of which produced boundary traces which corresponded to one of three composition planes.

From close examination of Fig 19, it may be seen that the twins seem to grow along the former slip lines.

By repolishing the specimens and

Table 1 . . . Analysis of Alignment of Twin Composition Planes with Slip Lines

	Br-2-T Angle between Axis and Slip Line		Br-3-T Angle between Axis and Slip Line		Br-4-T Angle between Axis and Slip Line	
	Measured* Degrees	Re- oriented† Degrees	Measured* Degrees	Re- oriented† Degrees	Measured* Degrees	Re- oriented† Degrees
Primary Slip Lines.....	40	24	31	36	38	28
Twin Comp. Plane.....	32		26		28	
Conjugate Slip Lines.....	57	57	18	22	27	22
Twin Comp. Planes.....	59		22		30	
Cross Slip Lines.....	10	10	27	33	9	9
Twin Comp. Planes.....	16		22		10	

* Angles determined from stereographic analyses
† Angles measured on metallograph

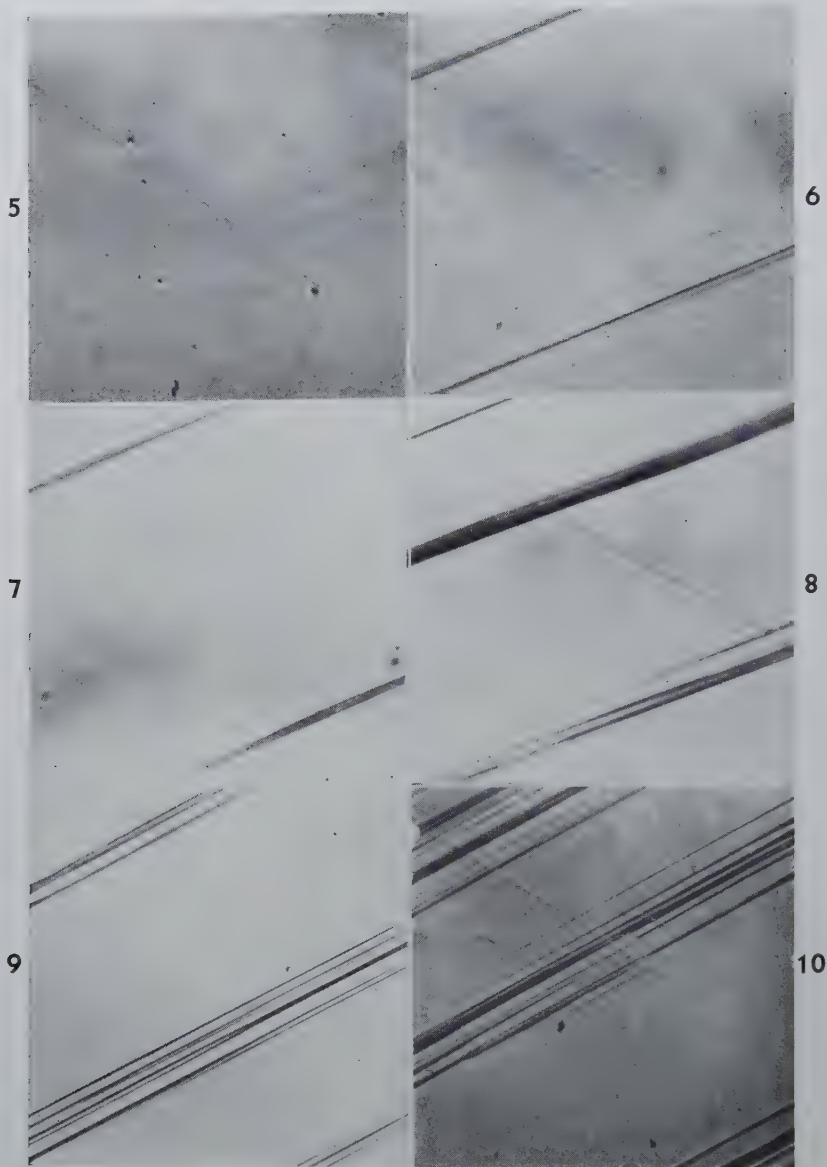


FIG 5-10—Slip lines in Specimen BR-2-T corresponding to various amounts of extension.

Except in the case of Fig 5, all micrographs were taken at a position around the conical surface of the specimen which would lie in a plane perpendicular to the plane containing the specimen axis and the primary slip direction. Stress axis vertical. Reduced one fifth in reproduction. $\times 500$

again straining in tension, slip lines were made to reappear in the unrecrystallized structure at the head of the taper and in the new grains. By aligning the composition planes of the twins with the slip lines in the parent lattice, that is, by moving the field from twin to original lattice along the axis of the specimen, these composition planes were identified as approximately parallel to former primary slip lines, cross-slip lines and conjugate slip lines. Several cases of close alignment are shown in Fig 21-26. In other cases, misalignments amounting to as much as 11° were found. Attempts were made to locate twins close to the unrecrys-

tallized structure so as to avoid misalignments due to the rotation of the lattice during the plastic extension preceding recrystallization. Since this was not always possible, stereographic analysis was used to correct the cases of misalignment by plotting the angles at the point of observation between the slip lines and the stress axis before and after straining. A typical construction is shown in Fig 27 from which the angles between the axis and the reoriented primary and cross slip planes respectively shown in column 3 of Table 1 were derived.

If observations of slip lines are made in the plane AP , then the trace of the

slip plane (primary) in the plane of observation is at the intersection of the two planes, viz., at P , and the angle AP , equal to 40° , is the measured angle between the axis and the trace of the slip plane.

If twins arising from this slip plane are observed in a part of the specimen in which the change of orientation during this extension is represented by moving the axis from A to A' , the angle $A'P$, equal to 24° will now represent the angle between the axis and their composition plane provided the latter occupies the new position of the slip plane with respect to the axis. In the case of the cross-slip plane, the

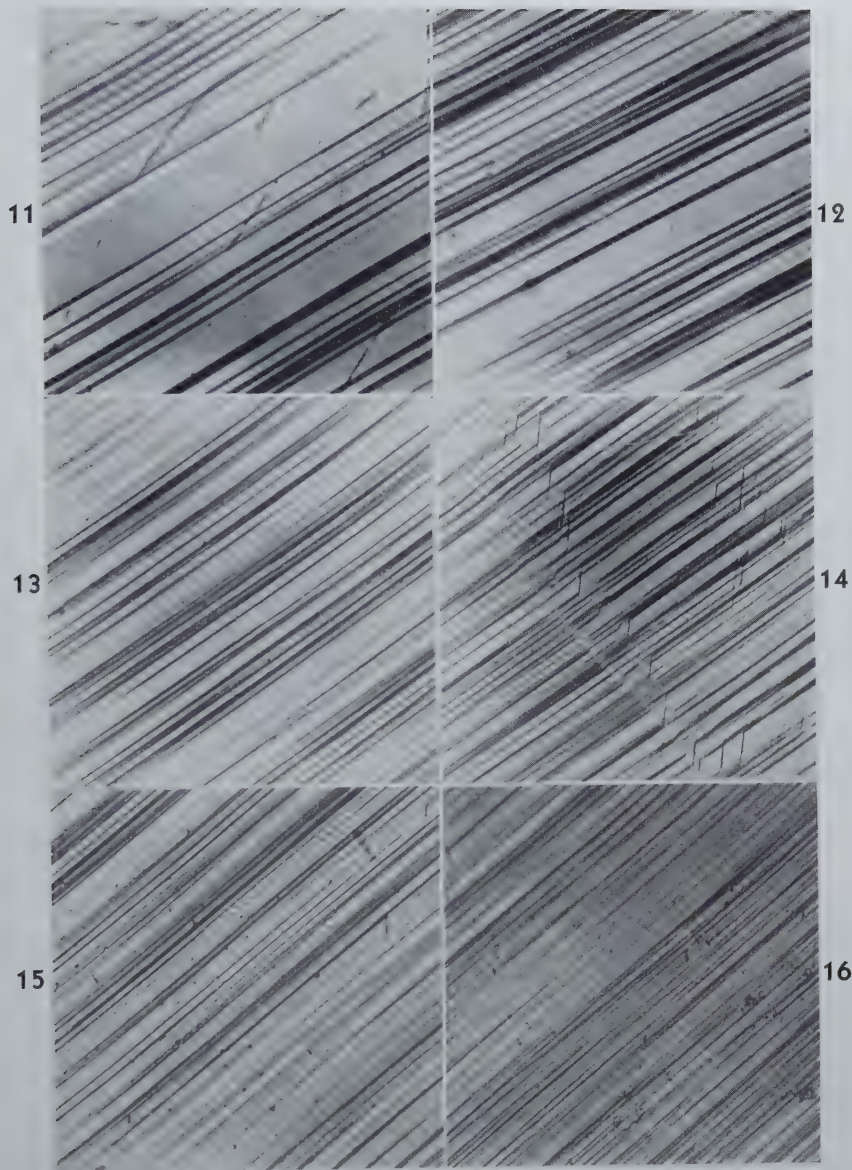


FIG 11-16—Slip lines in Specimen BR-2-T.

Positions similar to those represented in Fig 6-10. Stress axis vertical. Reduced one fifth in reproduction. $\times 500$

angle of its trace to the axis in the plane of observation AC , is equal to 10° and is practically unaltered in the new position $A'C$ after rotation. This is about 6° less than the measured angle between the axis and the twin composition plane. Similar constructions were made for the conjugate plane in this specimen and for all twinning planes in the other two specimens, BR-3-T and BR-4-T, as reported in Table 1.

The maximum error in alignment shown in Table 1 is 11° . There are four recognized sources of error in the technique used; (1) a possible 2° error in the X ray determinations from which changing positions of the stress axis were ascertained, (2) a possible 6° error introduced by the taper of the speci-

mens, (3) a possible error associated with the translation of the specimen on the microscope from the position of annealing twin to the position of the corresponding slip lines, and (4) an undetermined error in measuring the trace of the cross-slip lines because of their minute lengths. One or all of these possible errors may explain the observed deviations from perfect alignment.

Finally, the orientations of a number of grains in each specimen were determined by the Laue back-reflection technique. One group of these orientations together with the orientation of the parent lattice are shown in stereographic projection in Fig 28. These projections were drawn with the X ray beam at B , the center of the diagram,

that is, normal to the exposed grain surfaces. In the diagram, P refers to the parent lattice and the numbers refer to the specific grains outlined in Fig 29. In all of these recrystallized grains, there was an octahedral pole close to one of the octahedral poles of the parent lattice (ringed in the projection) as observed in the strained and annealed condition and in the neighborhood of the recrystallized grain, viz., at position 2 in Fig 29. It is doubtful if the term "recovered lattice" can be used in this connection since subsequent experiments have shown that the upper (least strained) part of a tapered specimen which showed no recrystallization at 800°C may recrystallize if annealed at a still higher temperature (900°C). In spite of the difference of



FIG 17—Appearance of first slip lines in Specimen BR-2-T.

Position perpendicular to the plane containing the specimen axis and the pole of the primary slip plane, that is, at the head of the major axis of the glide ellipse. Stress axis vertical. Note cross and conjugate slip lines. $\times 500$

orientation, which may amount to as much as 12° , between this octahedral plane as observed in the parent lattice and in the recrystallized grain, it is believed to be a common plane in the sense that it constituted the nucleus in the parent strained crystal from which the new grain grew, with a certain adjustment of orientation to the environment of strain, by a rotational process either identical with or akin to the one recently described by Kronberg and Wilson.⁶

The subsequent analytical technique was simply to perform the small rotation of the projection which was necessary to bring the so-called "common" octahedral pole in the recrystallized grain into exact coincidence with its position in the unrecrystallized structure, followed by the substantial rotation around this "common" pole which brought about complete coincidence of the two structures.

The results are shown in Table 2.

While this subject cannot be regarded as closed in view of the present meager understanding of lattice strain

in relation to recrystallization, the present experiments permit the following conclusions:

The recrystallized structure after substantial plastic deformation contains an octahedral plane obviously derived from a similar plane in the parent lattice.

The new grain does not grow out into the matrix without change of orientation or as an unreoriented twin. Its structure may be derived from the original structure by a rotation around the polar axis of the common octahedral plane.

Many of these rotations approximate the 22° and 38° rotations found by Kronberg and Wilson⁶ in their analysis of annealing twins in the secondary recrystallization of copper.

The rotation is presumably the response of the crystal to an activating strain which seems to be generally associated with twin faulting.

The Kronberg and Wilson analysis shows how a gross rotation of the lattice around an octahedral pole, for example, may be attained by small

coincidental movements of the atoms chiefly around sites which have retained their identity after the specific gross rotation. It is possible in this way to start with a twin fault and by thus moving the atoms around coincidence sites in neighboring planes on one side of the fault, to grow a new crystal whose octahedral planes are rotated 22° with respect to those of the original crystal, while growth on the other side of the fault by a similar process produces a twin structure whose poles are rotated 38° in the opposite direction around the composition plane. Other rotational possibilities are pointed out by Kronberg and Wilson. The present experiments are not wholly consistent with this crystal-geometrical pattern but offer strong evidence that the annealing twins in brass grow from twin faults parallel to the active slip planes by some rotational process of reorientation determined by the condition of strain in the plastically deformed crystal.

Inherent in the theory of two stage slip is the consideration that movement of the atoms in an octahedral slip plane determined by an active component of stress along the close packed strings of atoms $\langle 110 \rangle$ if forced to deviate into the interatomic valleys $\langle 112 \rangle$, (as shown by the arrows in Fig 1) which are assumed to be the paths of least resistance, would introduce a condition of residual stress in the hypothetical twin fault directing its alignment with the active component of shear stress. Thus the twin-faulted regions could be focal points of activation for a rotational process of atomic rearrangement and the observed annealing twins would be reoriented according to some pattern generally consistent with the reported observations.

Discussion of Results

Previous observations^{2,3,7,8,9} of lack of strain hardening in 70-30 brass single crystals during the early stages of deformation, are not in agreement with the present observations of strain hardening summarized in Fig 3. However, an explanation for these phenomena may be sought in the method of straining. In most of the experiments cited,^{2,3,7,8} the authors strained their crystals repeatedly but released the load after each individual extension. In the present investigation, the crystals attained their total extension

Table 2 . . . Rotation of Provisionally Reoriented Recrystallized Grains Around Designated Octahedral Poles Bringing Coincidence with the Original Structure

Specimen Number	Strain (Approx.* Axis Rotation Deg.)	Direction of Rotation	Rotation Deg.	Nearest Octahedral Pole of the Parent Crystal
BR-2-T	4			
	3	Counter clockwise	34	Cross slip plane
	5	Counter clockwise	24	Primary slip plane
BR-4-T	6	Counter clockwise	35	Primary slip plane
	5	Counter clockwise	18	Cross slip plane
	3	Counter clockwise	30	Cross slip plane
BR-3-T	1	Counter clockwise	18	Conjugate slip plane
	7	Counter clockwise	0	
	8.5	Counter clockwise	12	Cross slip plane
		Clockwise	24	Cross slip plane

* Determined from location of grain on the tapered specimen.



FIG 18—Three views of recrystallized portion of Specimen BR-2-T.
Slightly reduced in reproduction. $\times 1.5$



FIG 19—Three views of recrystallized portion of Specimen BR-4-T.
Slightly reduced in reproduction. $\times 2.5$

under one application of load. This points to the possibility that a recovery process may be responsible for the lack of strain hardening previously observed and that under continuous loading, the normal process of strain hardening occurs.

A significant observation from the resolved shear stress-shear curves of Fig 3 is the constant rate of strain hardening in all three orientations investigated, up to a value of shear of about 0.22. Beyond this strain the rate of hardening appears to be a function of orientation. And this value of shear was the minimum one at which recrystallization occurred at the selected annealing temperature (800°C). The exact significance of this shear value (0.22), that is, whether or not it is constant for all orientations at all temperatures of deformation and recrystallization must be determined by further study.

The composition planes of the annealing twins formed on recrystallization of the axially strained tapered crystals of 70–30 brass were always approximately parallel to one of the active slip systems. The orientation of a new grain or part of a twin could be derived from the orientation of the parent crystal in the strained condition by rotation around their “common”

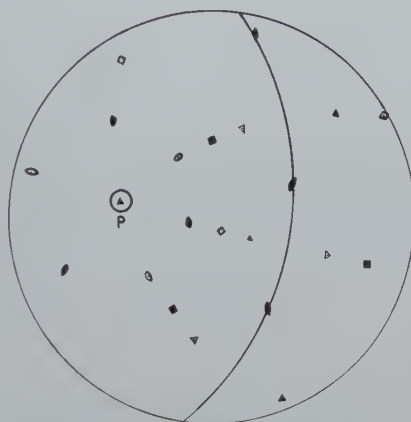


FIG 20—Stereographic plot of two sets of Laue spots showing the twin relation between the spots.

The twinning pole is labeled as *P* and its plane is shown as the arc in the plot.

octahedral pole, derived from the primary, cross or conjugate slip plane, but a certain small angular adjustment was necessary to bring the “common” pole into exact coincidence.

An attempt was made to rationalize the rotational reorientation by postulating a condition of residual stress at a twin fault due to the nature of the slip process. According to this view, the twin-fault is a preformed nucleus which activates a crystal-geometrical process of atom regrouping by rota-

tional shearing movements similar to those already reported by Kronberg and Wilson.⁶

In order to distinguish between the failure of the tapered specimens to recrystallize at 800°C in the regions which had been slightly deformed and their copious recrystallization in the severely strained regions, the following suggestions are offered on the basis of a two-stage slip process. Here a twin fault is produced in the first stage and a return to the original structure in the second stage (always accompanied by a certain configuration of residual stress).

In the early stage of slip, the atoms may be considered to move freely into their appropriate positions so as to complete the two $\langle 112 \rangle$ movements and result in an essentially unchanged lattice. However, during the later stages of the slip process, apparently beginning at about 0.22 shear, the lattice may be so strained or fragmented by the action of the three cooperating slip systems that the primary configuration can no longer be attained.

Summary

1. In the tensile straining of tapered 70–30 brass single crystals, variable de-

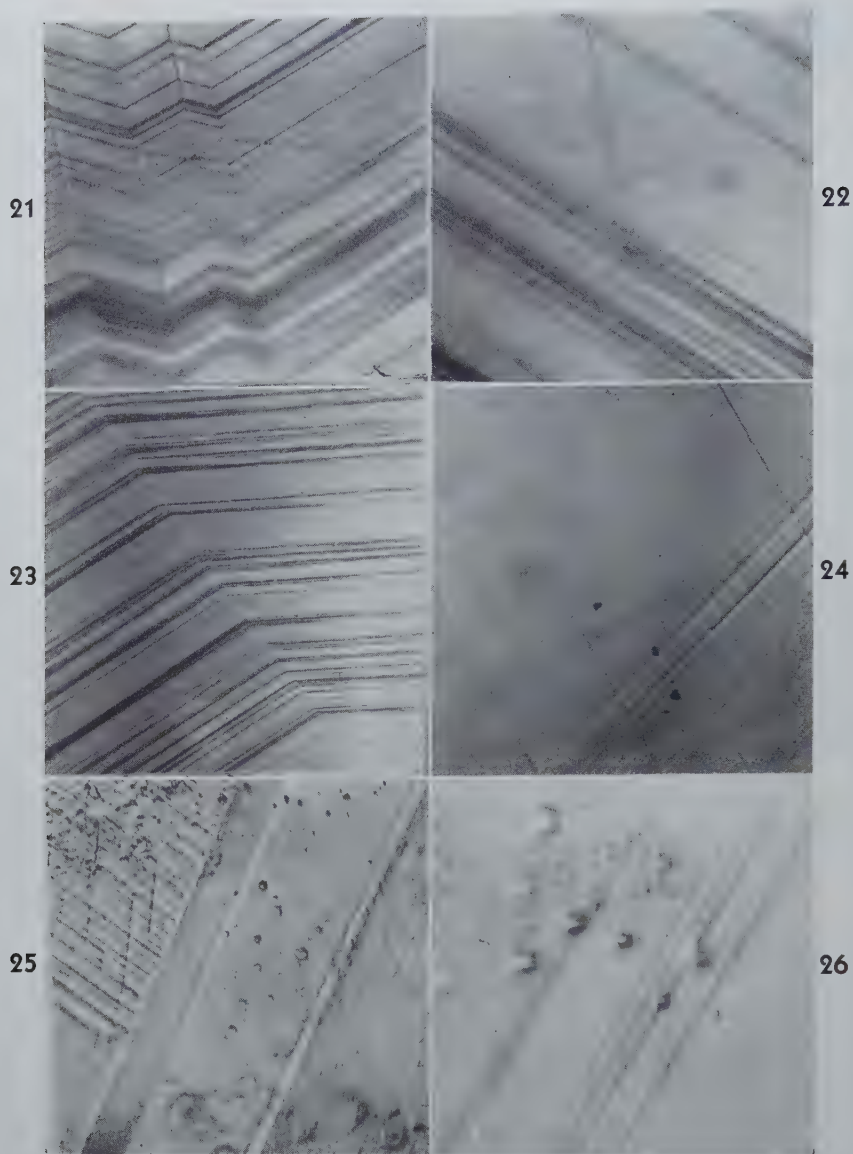


FIG 21-26—Alignment of composition planes of annealing twins with slip lines in the parent lattice.

Stress axis vertical. Reduced one fifth in reproduction. $\times 500$

formation along the specimen axis afforded the opportunity to record micrographically the progress of formation of slip lines.

2. Calculated resolved shear stress-shear curves show that there is slight strain hardening up to a value of shear amounting to about 0.22. At this point there is a marked rise in the rate of strain hardening which now appears to be a function of orientation.

3. Recrystallization on annealing at 800°C begins at a minimum value of shear of about 0.22.

4. Annealing twins are formed with composition planes parallel to the former operative primary, cross and conjugate slip planes, with no other relationships observable.

5. Recrystallized grains are found to

be related to the former parent lattice as reoriented after straining by a rotation about the pole of an operative primary, cross or conjugate slip plane. The variable amount and direction of rotation are apparently due to the difference in the strain configuration throughout the specimen.

These observations are interpreted as strong evidence in favor of coordinated $\langle 112 \rangle$ slip.

Acknowledgments

The authors' thanks are due to the New Jersey Zinc Co. of Pennsylvania for financial support which made this investigation possible and to the American Brass Co. for supplying the brass

from which the single crystals were grown.

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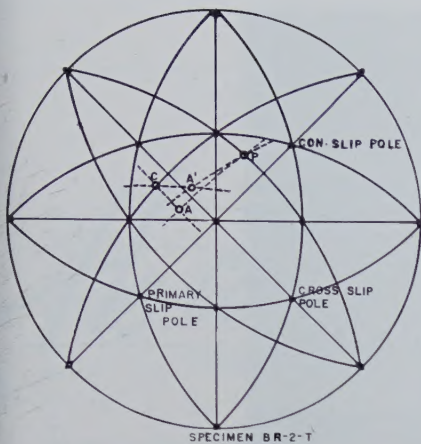


FIG 27—Stereographic analysis of the change in angle between slip lines and specimen axis at the point of observation before and after straining.

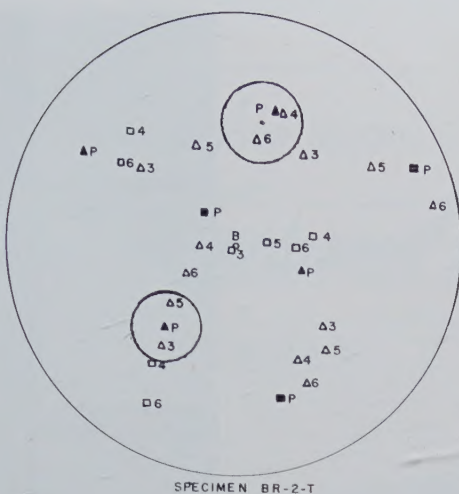


FIG 28—Stereographic plot of orientations of four grains in Specimen BR-2-T.

P refers to the parent structure and the numbers refer to the grains outlined in Fig 29.

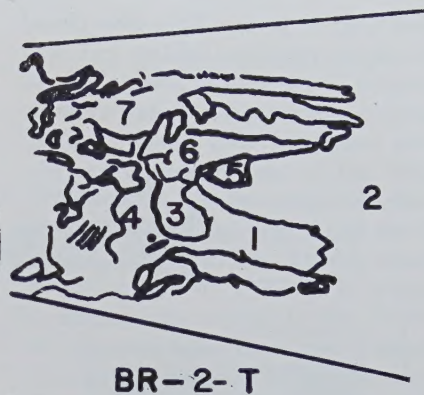


FIG 29—Outline of grains whose orientations are shown in Fig 28.

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Some Observations on the Rate of Secondary Recrystallization in High Purity Copper

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It is well known that if copper that has been severely cold-worked by rolling (70–98 pct) is heated to high temperatures a small number of large grains are formed at the expense of the fine grained structure that appears upon primary recrystallization.

Dahl and Pawlek,¹ Cook and MacQuarie,² Ward,³ and Boas and Bowles⁴ have studied this phenomenon in copper. It has been reported as taking place with measurable velocities in the temperature range 700–1080°. Boas and Bowles have reported that the time necessary for the transformation is of the order of days at 700°C. According to Ward³ the number of secondary grains which appear in OFHC

copper decrease with increasing perfection of cubical alignment after primary recrystallization; also the perfection of cubical alignment increases with decreasing grain size prior to the final reduction.

In this investigation two heats, A and B, of especially high purity copper (99.999 pct) supplied by the American Smelting and Refining Co., were used. The behavior of samples prepared from

the two heats was qualitatively very similar providing that the grain sizes established prior to the final reduction were about the same.

We observed that in specimens prepared from bars which were relatively coarse grained prior to final reduction secondary recrystallization took place at a rapid rate at temperatures as low as 500°C. This is about 200–300°C lower than the temperature necessary to obtain fairly rapid transformation in OFHC copper. A 0.22 in. thick rectangular bar of lot B was cold-reduced 60 pct by straight rolling and given a penultimate annealing treatment at 250°C. Following this it was cold-reduced 90 pct and heated to various temperatures in the range 450–1040°C. The specimens were about 8 mils thick

Technical Note No. 20 E. Manuscript received April 28, 1949.

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¹ References are at the end of the paper.

and $\frac{1}{2}$ sq. in. in area. They were placed in quartz specimen holders and heat treated in a hydrogen atmosphere. In general the secondary transformation was completed after about $\frac{1}{2}$ hr at 550° and in most specimens was partially complete after 6 hr at 500°C. From this it may be inferred that the linear growth rate in the transformation is at least 5×10^{-5} cm per sec at 500°C. Fig 1 shows photographs of typical specimens after heating for various times at the specified temperatures. Transformation is complete in all excepting those heated at 500°C. It is evident that the final grain size markedly increases with decreasing temperature so that at the lowest temperature only one grain appears per specimen.

Decreasing the penultimate reduction and increasing the penultimate annealing temperature tend to lower the temperature at which secondary recrystallization takes place rapidly. A sample of *B* given a penultimate reduction of 50 pct, penultimately annealed at 350°C, and cold-reduced 90 pct, exhibited complete recrystallization to several grains after 6 hr at 500°C.

In agreement with work on OFHC copper our results indicate that the tendency of secondary grains to form decreases with increasing perfection of cubical alignment. However, we have observed that the rate of appearance of secondary grains in specimens having a high degree of cubical alignment is greatly accelerated by very small strains. Specimens were prepared from heat *A* as follows: a 0.164 in. thick rectangular bar was cross-rolled to a reduction of 50 pct and annealed at 400°C for 3 hr. Because of the smaller initial grain size the grain size resulting from this treatment was much less than that obtained in lot *B* after the penultimate annealing treatments described above. The bar was cold-reduced 90 pct by straight rolling and cut into specimens having the same size as those from lot *B*. Rapid heating of these specimens to 1040°C and holding for 4 hr produced complete secondary recrystallization to 1 or 2 grains per sq. in. However, specimens recrystallized by heating at 500°C for one hour or heated slowly (200°C per hr) showed no evidence of beginning secondary recrystallization after periods of 16 to 20 hr at 1040°C provided that care was taken not to strain the specimens after primary recrystallization. If the specimens were strained by small amounts after primary recrystallization

the secondary transformation was complete in nearly all specimens after 4–6 hr at 1040°C. Strains as small as 0.085 pct maximum, produced by bending the specimen over cylinders of a given radius of curvature, were sufficient to cause this effect. These results are in agreement with those obtained independently by Kronberg and Wilson⁵ on OFHC copper although they apparently used much larger strains to accelerate the secondary transformation.

The orientations of the secondary crystals which we observe are in fair agreement with those reported for copper by Boas and Bowles.

Our results demonstrate that the rate of the secondary transformation is much more rapid in high purity copper than in OFHC copper. At 500°C the rate of growth of secondary grains in high purity copper is at least 10^5 times greater than the value obtained for OFHC copper by extrapolation of Ward's results. Also it may be concluded that the secondary transforma-

tion takes place very slowly if at all in specimens exhibiting a high degree of perfection in cubical alignment. Secondary transformation is accelerated (or perhaps initiated) in the latter specimens by strains as small as 0.085 pct. The orientation of the grains thus obtained is very near that of the grains appearing in the secondary transformation of samples rapidly heated to the transformation temperature from the cold-rolled state. These results will be reported more completely in a subsequent paper.

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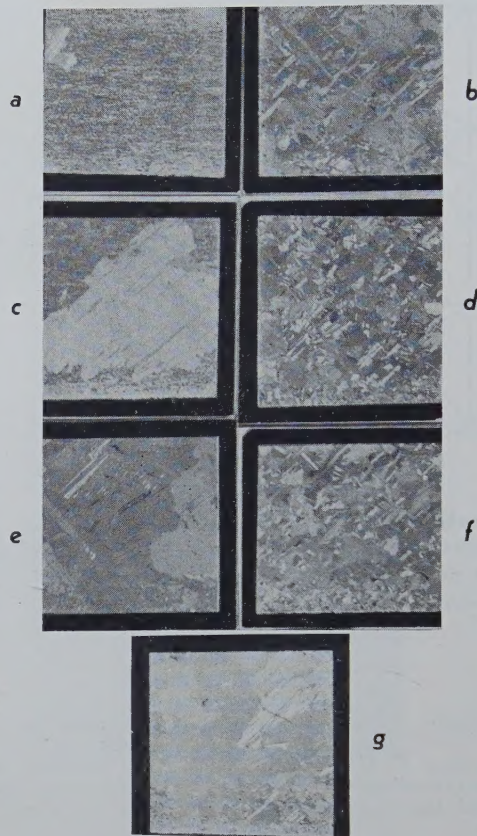


FIG 1—Copper samples annealed at various temperatures after 90 pct reduction.

a. 500°C—1 hr. b. 750°C—1 hr. c. 500°C—6 hr. d. 900°C—1 hr. e. 550°C— $\frac{1}{2}$ hr. f. 1040°C—1 hr. g. 600°C—5 min.

ALARM INDICATOR RECORD

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TIME RECEIVED	A	P	BY	TRouble FOUND
SENDING OFFICE				
RECEIVING OFFICE				
DATE OK				TIME A P BY

SYNCHRONIZATION - START						CA FAIL SEND CKT.		STATION IDENTIFICATION			
ON	ON	OFF	ON	ON	CHAN. 1 SECT						
SYNCHRONIZATION - STOP						FUEL GAS LOW					
ON	ON	OFF	ON	ON							
FUSES		24-VOLTS		ABS							
DISCH.	DIST.	H-L VOLT	REG. FAIL	24V 130V	48V						
POWER CONTROL PANEL FAILURE											
201-202W	203-204W	205-206W	207-208W	201-202E	203-204E						
ALT. CONT. BAY - NO VOLT. OUT.					NO VOLT. - TRAN						
201 202	203 204	205 206	207 208	201 202	203 204						
RECT. FAIL 24/130V		48 V H-L VOLT		RECTIFIER - INVERTER FAIL							
				NO. 1 NO. 2 NO. 3							
64 KC PILOT ALARM AT NON-SW. MAIN								3096 (WKG. LINE) PILOT AT SW. MAIN			
201	202	203	204	205	206	207	208	201 203	205 207	202 204	206 208
2064 KC PILOT ALARM AT NON-SW. MAIN								3096 (SP. LINE) PILOT AT SW. MAIN			
201	202	203	204	205	206	207	208	201 203	205 207	202 204	206 208
3096 KC PILOT ALARM AT NON-SW. MAIN								SP. LINE FAIL AT SW. MAIN			
201	202	203	204	205	206	207	208	201 203	205 207	202 204	206 208
TOT. LINE FAIL AT SW. MAIN				AUTO. SWITCH AT SW. MAIN				AUTO. SW. LOCKED AT SW. MAIN			
201 203	205 207	202 204	206 208	201 203	205 207	202 204	206 208	201 203	205 207	202 204	206 208

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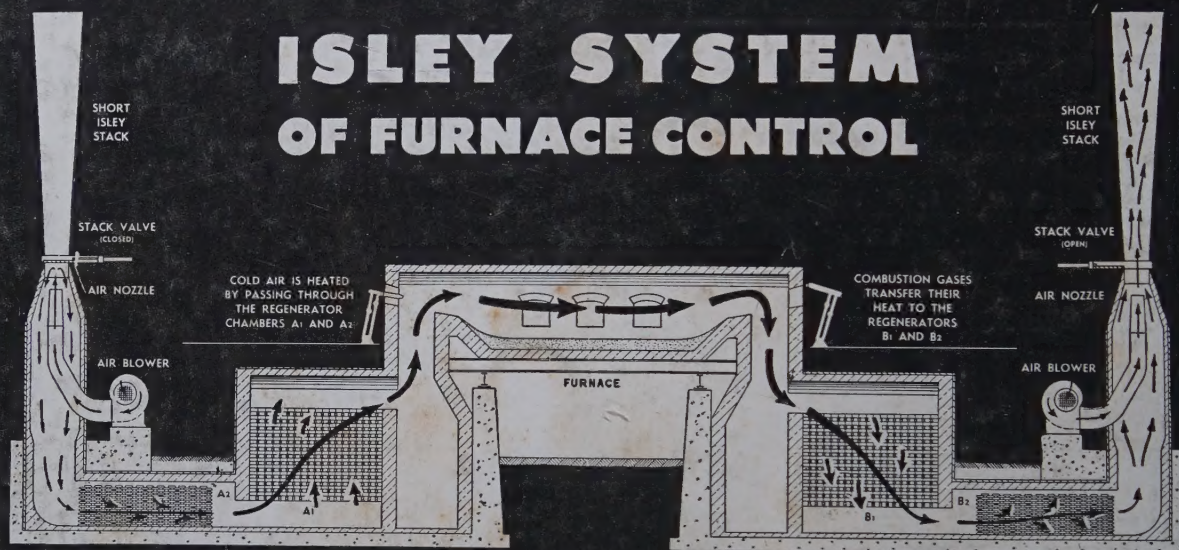
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